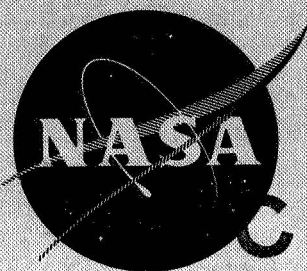


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DETERMINING THE FEASIBILITY OF CHEMICAL
VAPOR DEPOSITION PROCESS FOR THE PRODUCTION
OF DISPERSOID STRENGTHENED CHROMIUM ALLOYS

by

Gene F. Wakefield and Richard D. Humphries

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NAS 3-10488

TEXAS INSTRUMENTS INCORPORATED
Materials and Services Group
Chemical Materials Division
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FINAL REPORT

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November 25, 1968

CONTRACT NAS 3-10488

Technical Management
Lewis Research Center
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FORWORD

The research described herein, which was conducted by Texas Instruments Incorporated, Chemical Materials Division, was performed under NASA Contract NAS 3-10488. The work was done under the management of the NASA Project Manager, Mr. John P. Merutka, Air-Breathing Engine Procurement Section, NASA-Lewis Research Center. This report is also issued under Texas Instruments' Rpt. No. 04-68-12.

ABSTRACT

A chemical vapor deposition process was developed for preparation of chromium metal in dense, bulk form. Incorporation of a dispersoid phase of 0.01-micron thoria was only partially successful due to difficulties in feeding the fine powder. The chromium metal was higher purity and showed some ductility. Thermal treatment at 2600°F resulted in recrystallization and some reaction of chromium oxide inclusions with the matrix.

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I. SUMMARY

The objective of this program was to develop a process and a system for producing high-purity chromium metal containing a thorium-oxide dispersion material. The program consisted of two main tasks:

- Develop a laboratory apparatus for chemical vapor deposition of high-purity chromium metal in a dense bulk form; this entailed equipment design, assembly, testing, and subsequent development of parameters influencing deposition of pure chromium in rod form
- Adapt the system to the introduction of thorium-oxide powder during chromium deposition in order to produce a suitable dispersion material

Though considerable difficulty was encountered in producing dense bulk deposits of chromium, 3/8-in. dia x 4-in. length deposits could be made.

Chemical and emission spectrographic analysis and electron micrography showed the chromium to be of high purity. This was supported by hardness values and the metal's cold-working characteristics. Hardness values ranged from 120 to 160 KHN (100-gm load); a 50 percent reduction in thickness could be achieved with only slight cracking around the edges of the specimen.

The addition of the dispersoid in powder form presented several problems. Controlled, uniform delivery of the thorium oxide to the deposition zone sometimes was not possible or was erratic because of plugging or packing of the powder in the delivery lines. The problem was somewhat improved but not completely resolved by rearranging the feed lines and gas flows.

Although considerable powder was brought into contact with the chromium deposit in later experiments, only a small amount in most cases could be detected in the chromium. Other experiments resulted in a low-density material containing agglomerations of ThO_2 in Cr.

II. INTRODUCTION

The increased performance demanded of mechanical systems invariably results in corresponding demands for materials with improved properties. In some instances, conventional metallurgical techniques and modest improvements have been sufficient to satisfy the property requirements. It is significant, however, that the major developments of new material systems have been the products of a new technology or of adaptations of a technology not common to the particular material field. Notable examples are boron filaments produced by vapor deposition, TD nickel, and synthetic-diamond production. Thus, attainment of a significantly improved material may well depend on use of new, less "normal" technologies possessing the specifically required attributes.

Improved materials are required to meet the demands for turbine buckets and vanes of advanced jet engines. Additional research and development are required for processes to provide dispersion-strengthened chromium alloys. It is important to determine whether the dispersion-strengthening approach (analogous to SAP or TD nickel) will provide a chromium alloy with strengths comparable to those of the better melted alloys but with improved low-temperature ductility.

The stress rupture strength targets of these materials for buckets and vanes are, respectively, 15,000 psi in 3000 hr at 2100°F and 4000 psi in 3000 hr at 2400°F. The targets for transition temperature are: in short-time tension, less than room temperature; in impact, less than 300°F. Protective coatings are envisioned as a possible means of protecting these materials.

The potential ability of chromium-based materials to meet these goals is well-known; additionally, chromium has some inherent corrosion resistance. The ultimate goals have been difficult to meet, however, because of the inherent limitations of conventional techniques for material systems, such as chromium and chromium alloys, which are sensitive to interstitial impurities.

Chemical vapor deposition (CVD) techniques routinely control detrimental impurities to very low levels, so application of this technology to metallurgical systems could provide the specifically required attributes.

A first step in achieving a suitable material is selection and/or development of a process that will result in the desired ultra-fine particle size and spacing of the dispersed phase. Furthermore, to achieve long-time strength at high temperatures, the dispersed phase must be stable. Previous studies show that instability of dispersed oxide phases in metal matrices is associated with impurities; thus, the process must be capable of producing the desired dispersed phase in a high-purity matrix.

This program was designed to demonstrate the feasibility of chemical vapor deposition technology for production of chromium of sufficient purity to have desirable mechanical properties and to introduce a dispersoid uniformly into the metal. Program success would indicate that CVD offers the potential of producing a whole new family of improved materials.

This program represents a unique extension of CVD applications based on production techniques now being utilized. The basic technology of growing bulk material through CVD has been in use for more than 10 years in production of silicon semiconductor material.

The capability of CVD processes to build material systems on the molecular level, i. e., one atom at a time, means CVD inherently has the following unique characteristics:

- Theoretical density can be attained in solid billet shapes during the growing phase, eliminating the necessity for secondary operations for the purpose of increasing density
- The ability to provide precise parameter control gives the technique versatility in deposition rates, structure, and alloy combinations
- Purity associated with chemical vapor deposition is routinely maintained in the low ppm range

In addition to these unique capabilities, the adaptability of CVD processing techniques to scale-up and large-scale production is significant to this program and has been amply demonstrated in the semiconductor industry.

The program was conducted in four phases:

- Phase I — equipment design, development, and preliminary operation
- Phase II — determination of parameters influencing deposition of pure chromium metal in rod form
- Phase III — investigation of the formation of the desired microstructure in the chromium matrix
- Phase IV — analysis to determine the product's microstructure, purity, and stability

III. PHASE I, EQUIPMENT DESIGN AND DEVELOPMENT

A. REACTOR DESIGN

The initial reaction-chamber design, shown in Figure 1, consisted of two concentric quartz tubes held in place by O-ring sealed metal end caps. In the chamber between the two tubes was a charge of chromium

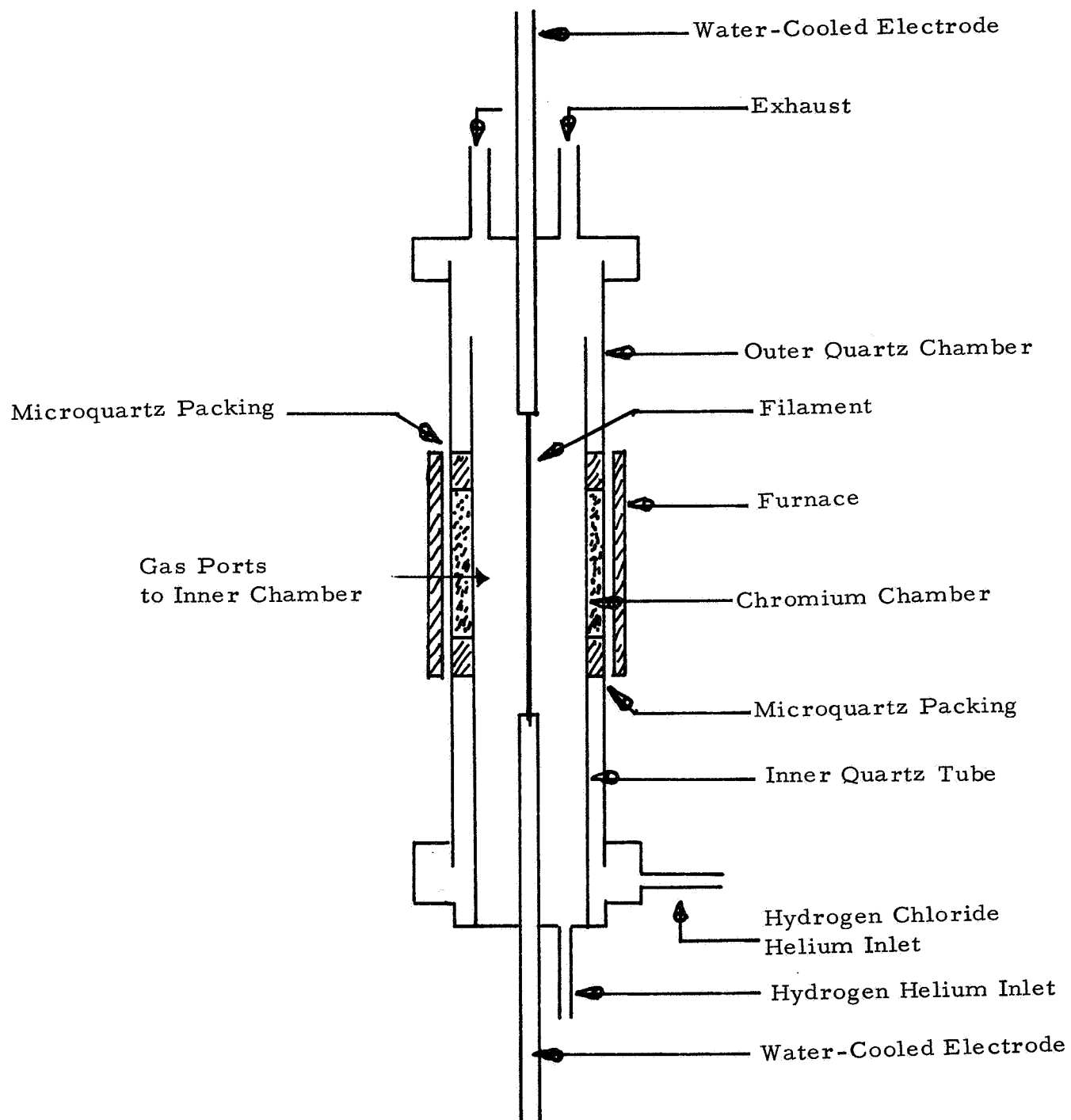


Figure 1. Schematic Diagram of Reactor

held in place by microquartz packing. The inner chamber contained a heated tungsten filament as a deposition site. Gas inlets were provided on the end caps so the appropriate gases could be introduced into the desired chambers.

A mixture of semiconductor grade HCl and purified helium was passed over the chromium (heated to 900° to 1000°C) to form CrCl_2 . After passing into the inner chamber through the ports shown in Figure 1, the CrCl_2 and helium mixture was blended with hydrogen; reduction to chromium was to occur on the tungsten filament.

Water-cooled electrodes were provided to prevent excessive heating of electrodes and end caps.

Figure 2 shows the reactor in operation.

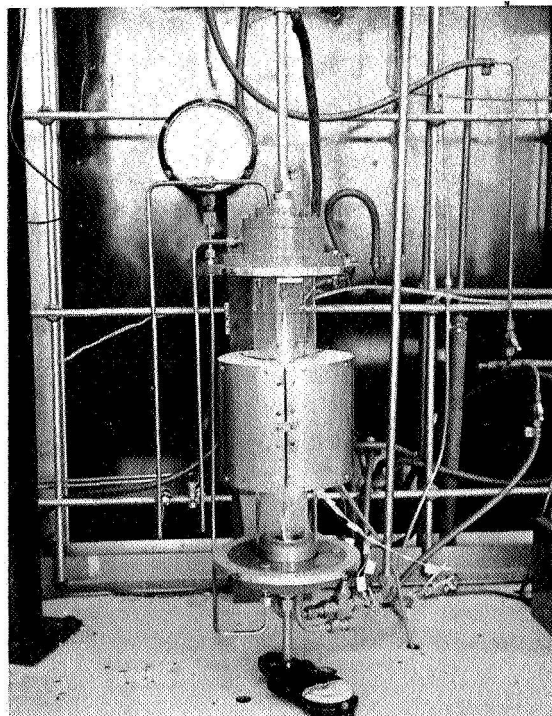


Figure 2. Reactor in Operation

B. ELECTRICAL SYSTEM

The electrical system sketched in Figure 3, consisted of a recorder controller in connection with a saturable core reactor and stepdown transformer arrangement to control filament temperature. In initial runs, an optical pyrometer was used to observe filament temperature. Control of the outer furnace temperature was monitored and recorded.

Figure 4 is a photograph of the filament controller, and Figure 5 is a photograph of the gas cabinet containing the furnace controller.

C. GAS SYSTEM

Figure 6 is a diagram of the gas-handling system.

Helium was purified by passage through a bed of titanium chips held at 700°C. Hydrogen was purified by a palladium diffuser. Hydrogen chloride was used without purification. Ball type flowmeters were used to measure gas flow rates.

D. STARTING MATERIALS

The following is a list of starting materials obtained and their manufacturings:

Chromium metal	United Mineral Corp.
Tungsten rods (1/8-in. dia x 18-in. length)	Phillips Elmet Corp.
ThO ₂ powder (0.01 to 0.07 μ)	Vitro Laboratories

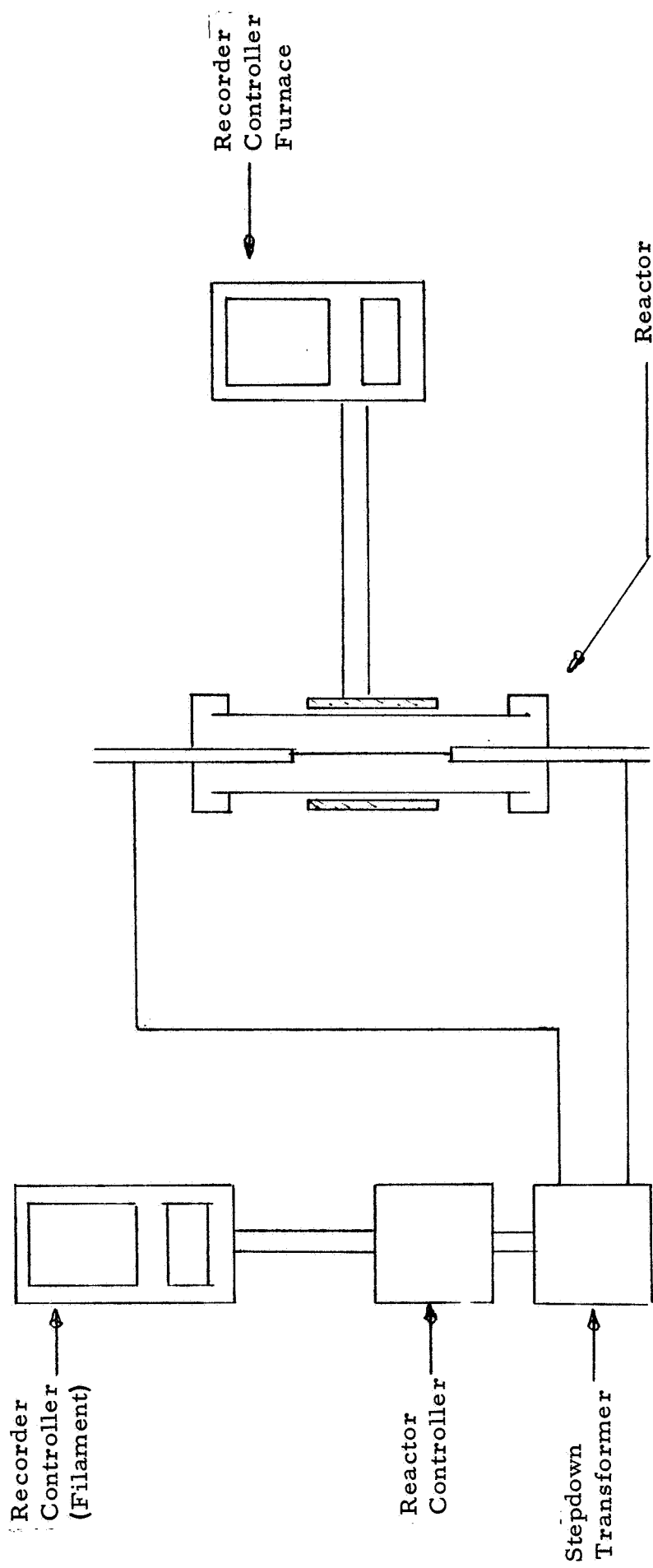


Figure 3. Diagram of Electrical System

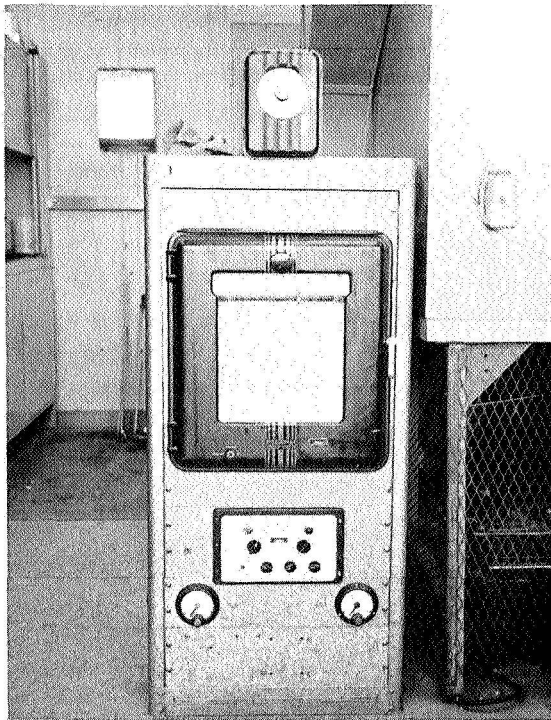


Figure 4. Recorder Controller for Filament Temperature

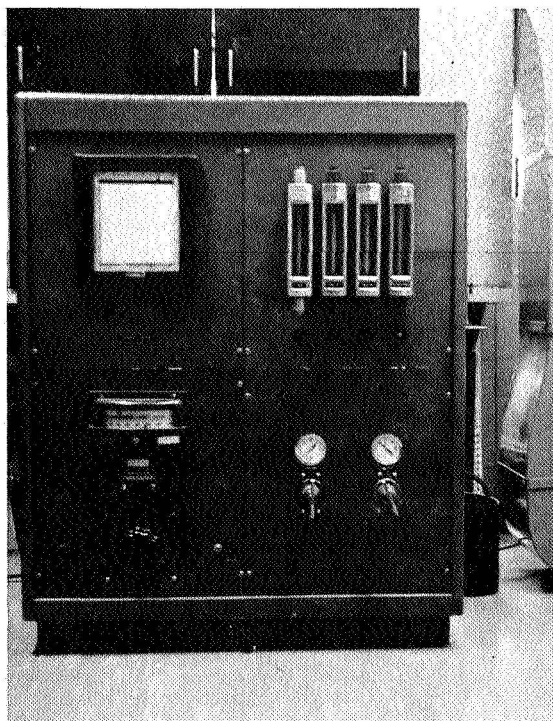


Figure 5. Control Cabinet Containing Gas-Flow System and Furnace Controller

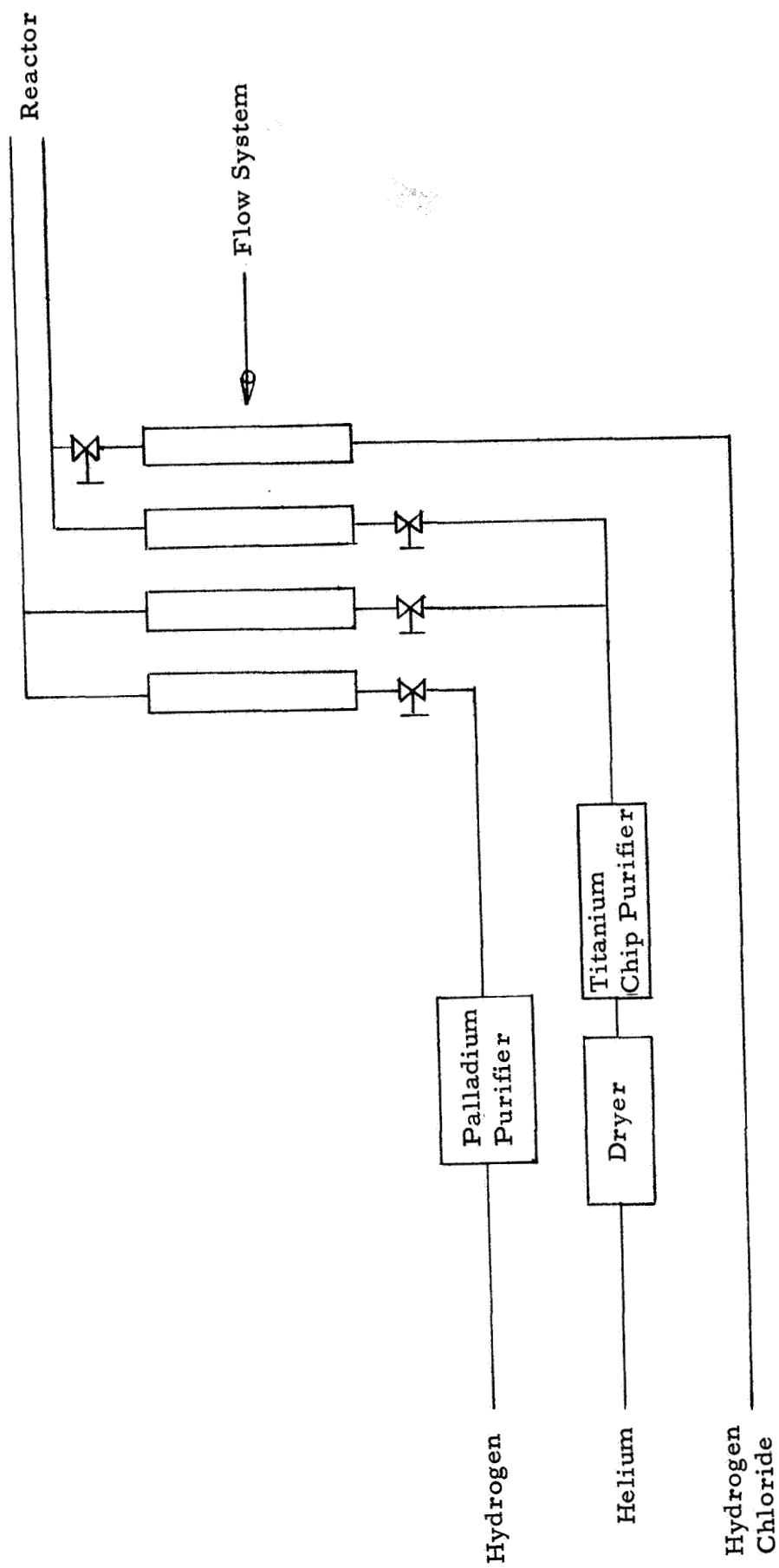


Figure 6. Schematic Diagram of Feed System

E. PRELIMINARY EXPERIMENTS AND DESIGN MODIFICATIONS

Seven experiments were performed to observe the system's controllability and to determine the necessary design changes.* From these runs, only a light (if any) coating of chromium resulted. Immediately apparent were problems with proper gas-mixing delivery to the deposition site and deterioration of quartz tubes in contact with liquid CrCl_2 . Although a sufficient amount of CrCl_2 was generated during the run, most of it remained in the generating chamber and, as a result, reacted with the quartz tubes.

To alleviate some of these problems, tube-design changes as shown in Figures 7, 8, 9, and 10, and gas-flow changes as indicated in Table 1 were made during these experiments. As shown in Figure 7 (initial tube design), four sets of four gas ports were used. No deposit resulted from two experiments performed in this tube. Light coatings and needle growths resulted when the tubes shown in Figures 8, 9, and 10 were used. Filament and furnace temperatures were not changed so that the effect of flow and tube design could be observed.

Variations in gas flow rate, direction of flow, and chromium-bed temperature were investigated in experiments 8 through 11. With no significant improvement in reactor performance, experiments 12 through 19 were run using Cl_2 instead of HCl ; the purpose was to determine whether the pressure of hydrogen from the $2\text{HCl} + \text{Cr} \longrightarrow \text{CrCl}_2 + \text{H}_2$ reaction was inhibiting transfer of the chromium chloride. This reaction could potentially form some CrCl_3 as well as CrCl_2 . Since CrCl_3 has a significantly higher vapor pressure and melting point, higher concentrations of chromium chloride could be moved to the filament and reaction of CrCl_2 with quartz would be minimized if the CrCl_3 were not reduced to CrCl_2 . A chromium deposit usually appeared on the reactor wall, but no significant coating appeared on the filament.

* Table I, at the end of this report, summarizes reaction conditions and results.

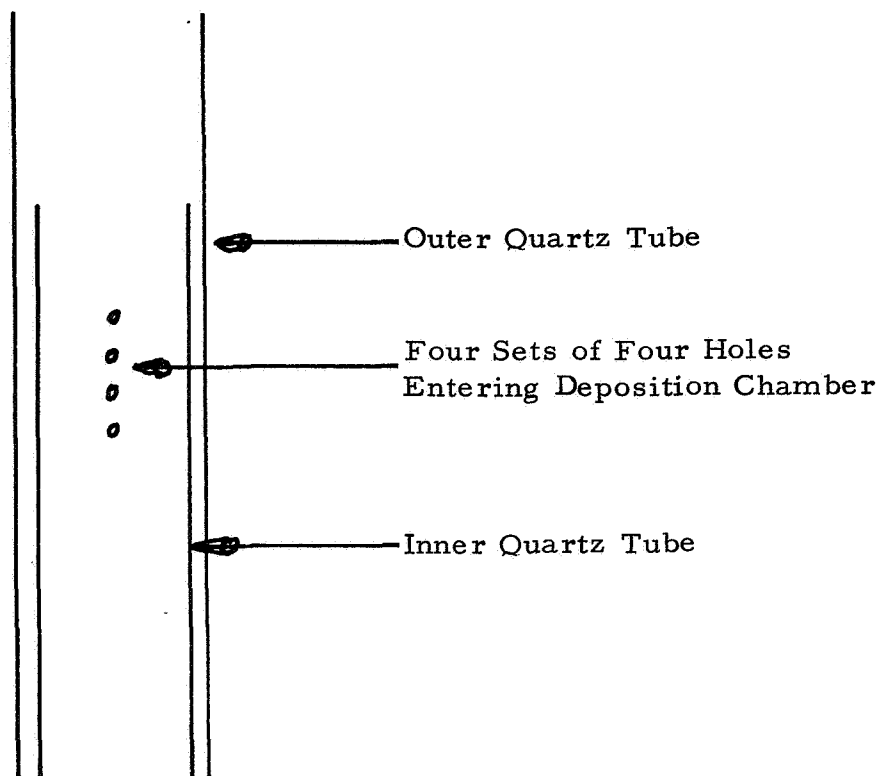


Figure 7. Initial Tube Design

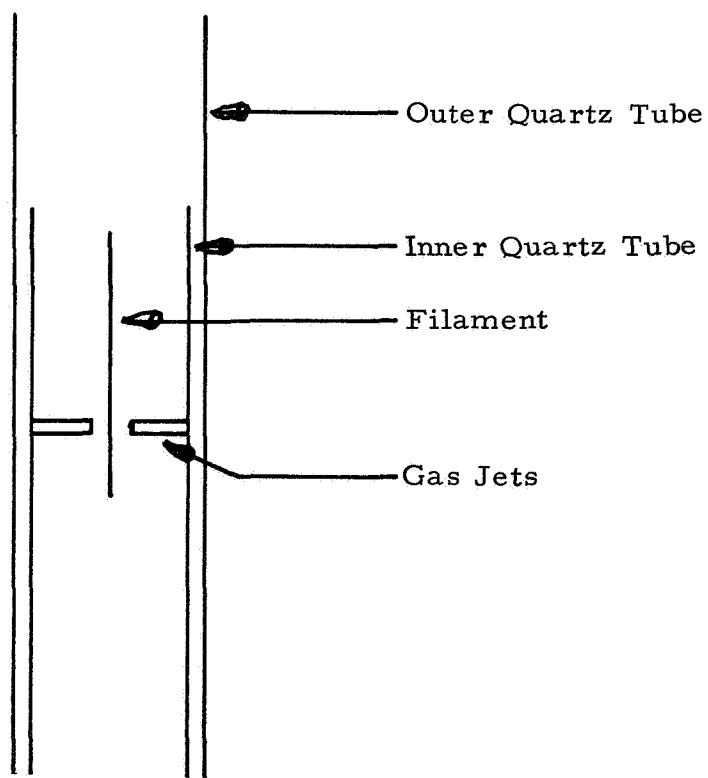


Figure 8. Second Tube Design

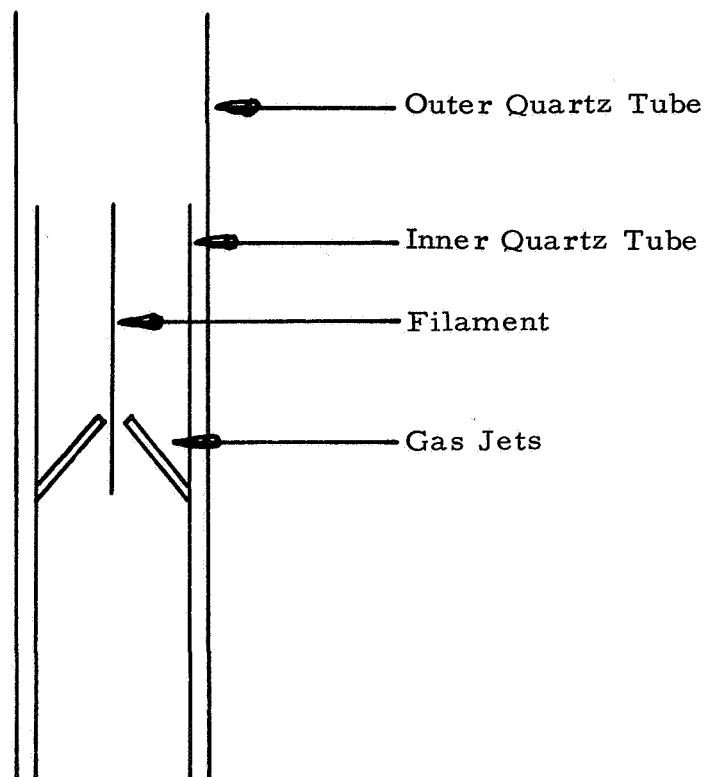


Figure 9. Third Tube Design

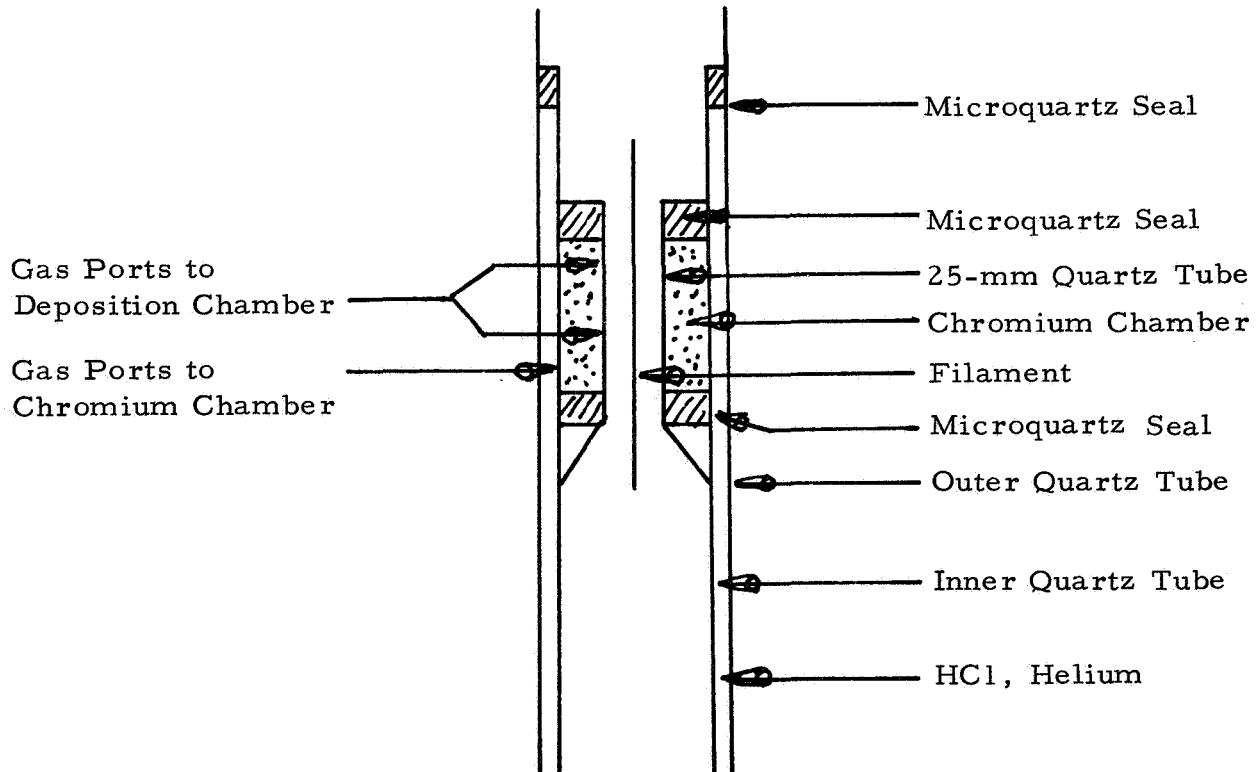


Figure 10. Fourth Tube Design

A small quantity of anhydrous CrCl_3 was obtained from Fisher Scientific Company to continue the experiments in which Cl_2 was used to form CrCl_3 as well as CrCl_2 . With this material, seven experiments (25 through 31) were performed in the reaction chamber shown in Figure 11. In experiment 25, a very light coating appeared at the lower end of the filament; experiments 26 and 27 were performed at higher furnace temperatures in an effort to increase the flow of CrCl_3 into the deposition chamber, but only a light coating resulted from one of these experiments. Experiments 28 through 31 were done with a small flow of chlorine mixed with the helium carrier gas to prevent reduction of CrCl_3 to CrCl_2 before it entered the deposition chamber. Although CrCl_3 could be moved into the deposition chamber, no significant deposit was obtained.

Because of the difficulties experienced in generating and controlling delivery of chromium chloride to the deposition chamber, the reactor design was modified as shown in Figure 12. In this system, chromium chips were contained in a quartz tube suspended from the top plate. CrCl_2 passed from the generating chamber into the deposition chamber through gas ports in the bottom of the tube. A thermocouple was extended into the chromium bed for better monitoring of the bed temperature. Hydrogen was mixed with the CrCl_2 approximately 1 in. below the CrCl_2 entrance ports.

This modified system was used for experiments 32 through 46. In experiments 32 - 35 no deposits of metal were obtained; however, in all other experiments deposits were made. Figure 13 shows the smooth coating of chromium obtained in experiment 36. Experiment 37 resulted in a lighter but smoother coating as shown in Figure 14, and experiment 39 produced a bulky but nodular deposit as shown in Figure 15. Experiments 36 and 39 produced 2- and 3-gm/hr deposits.

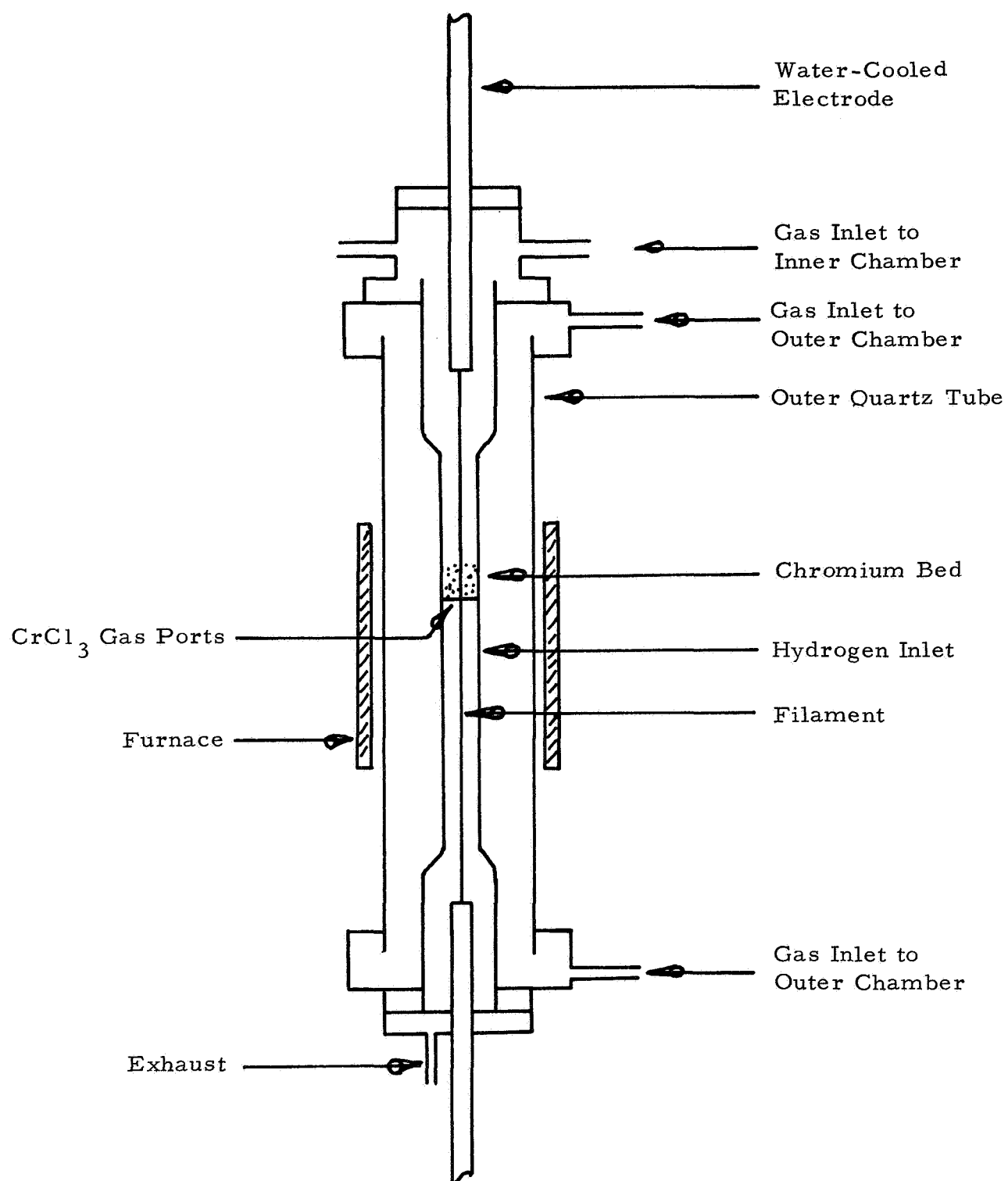


Figure 11. Reactor Design

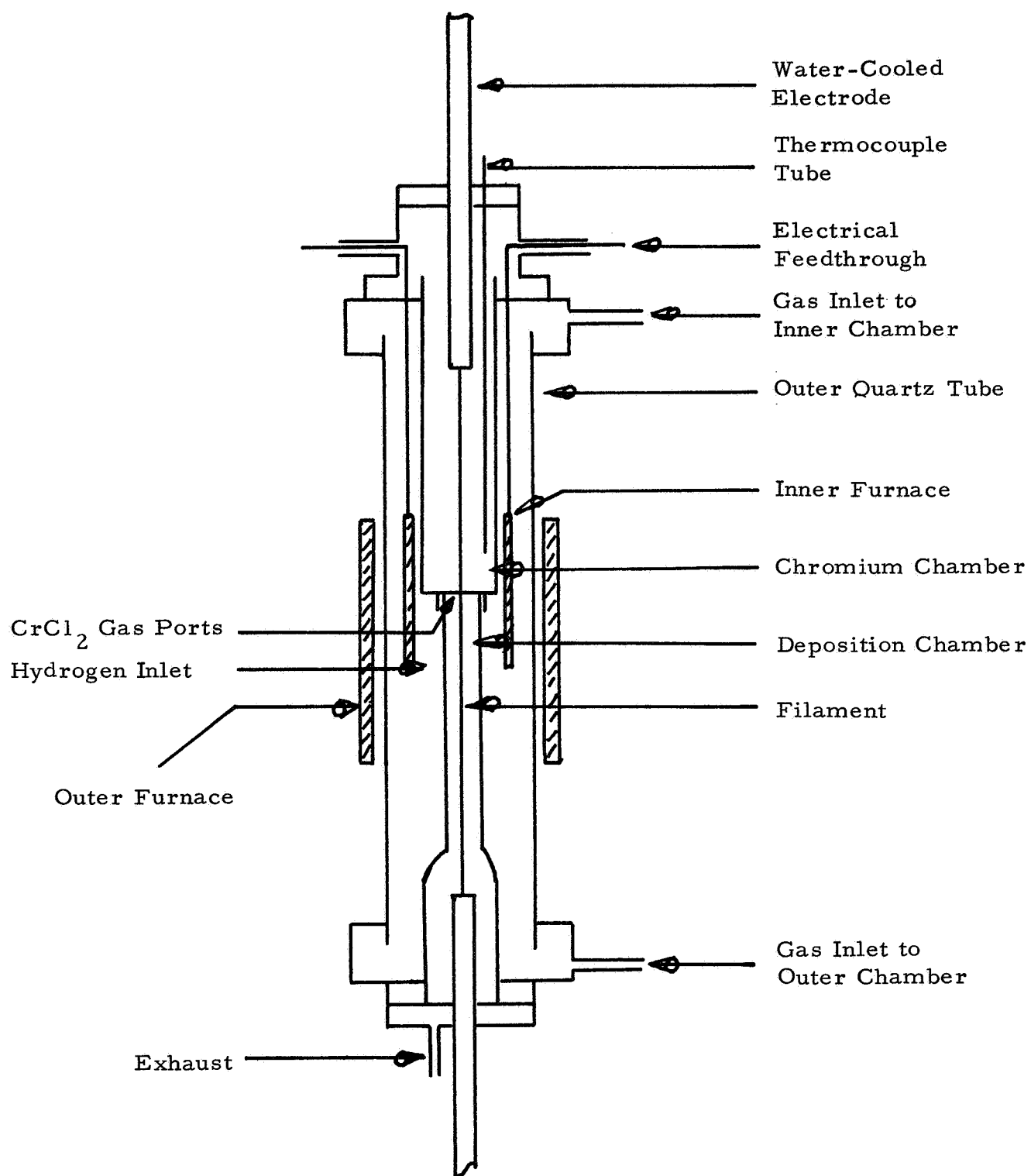


Figure 12. Modified Reactor Design

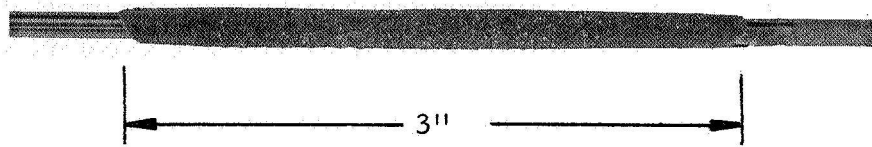


Figure 13. Chromium Deposit, Experiment 36

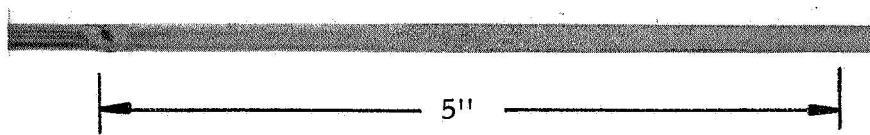


Figure 14. Chromium Deposit, Experiment 37

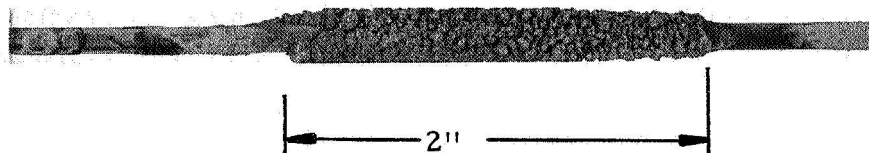


Figure 15. Chromium Deposit, Experiment 39

Gas compositions and flow rates were altered in experiments 40 through 46 in an effort to obtain a more uniform coating and to maintain a reasonable deposition rate. Smooth deposits resulted from experiments 40, 44, 45, and 46, although deposition rates were lower than in some other experiments. Rough and bulky deposits resulted from experiments 42 and 43 because CrCl_2 liquid ran down the filament.

IV. PHASE II, PARAMETER INFLUENCING DEPOSITION OF PURE CHROMIUM

The reactor design shown in Figure 16 was used throughout the remainder of the program with only slight modification. In later experiments, molybdenum shields were used in the generating and deposition chambers to extend the life of the quartz tubes exposed to the liquid CrCl_2 . One inside furnace, which was necessary in earlier experiments to augment the outside-furnace capacity, was later removed and replaced with a larger external furnace.

Phase II included experiments 47 through 91; many parameters were investigated. In experiments 47 through 70, combinations of gas flow rates, concentrations, and operating temperatures were studied. Though rates of deposition increased, large numbers of needles or large crystals appeared on the upper region of the filament. Typical crystal growths are shown in Figures 17 and 18 (photographs of experiments 55 and 56).

The best set of run conditions from this series was obtained from experiment 69. An increase in flow rates and furnace temperature and a decrease in filament temperature resulted in a smoother, more uniform deposit at a slightly higher deposition rate. Figure 19 is a photograph of experiment 69.

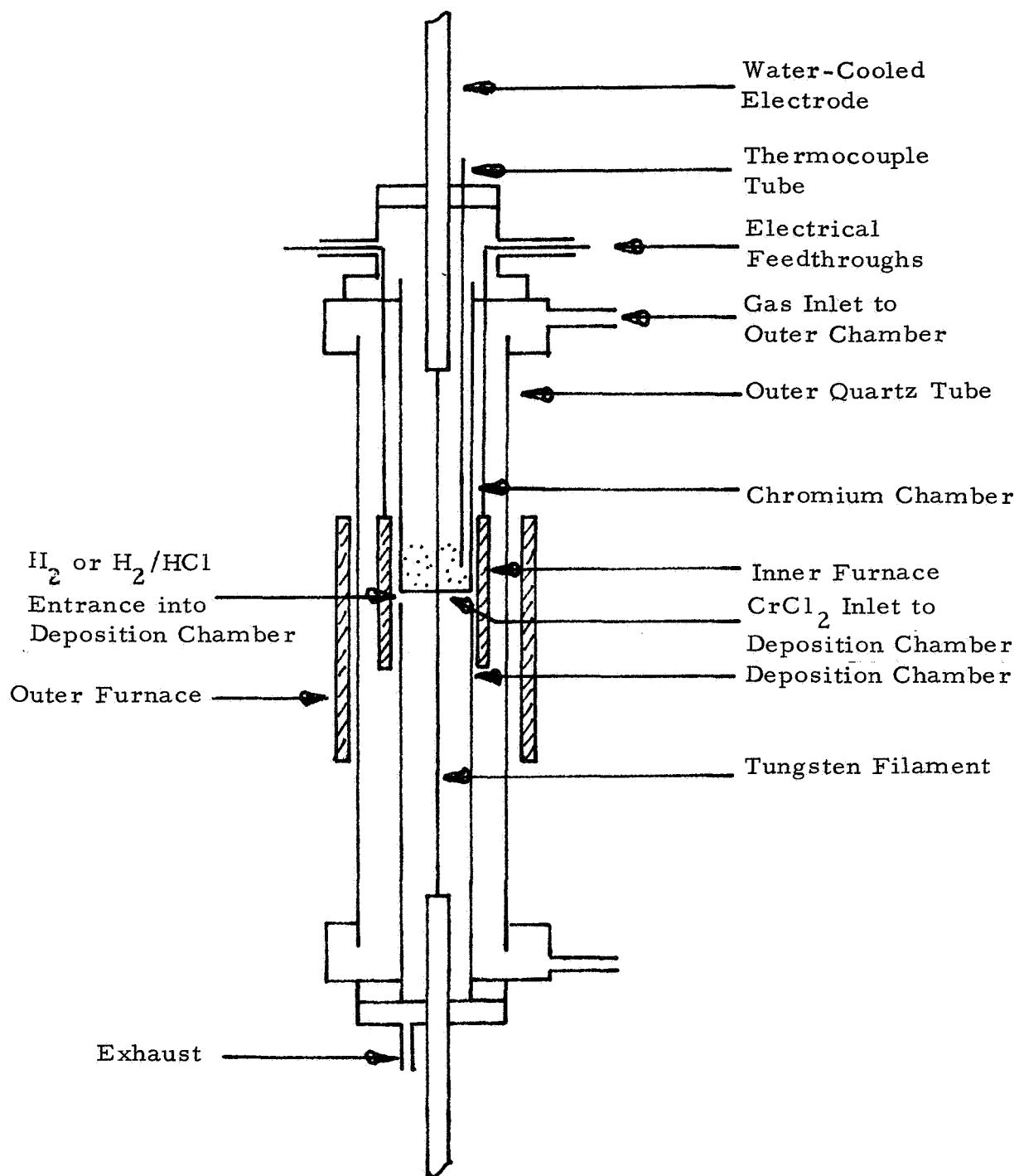
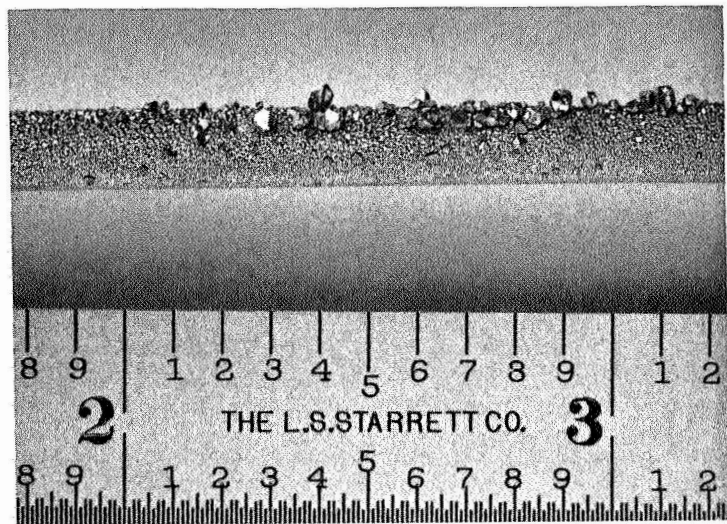
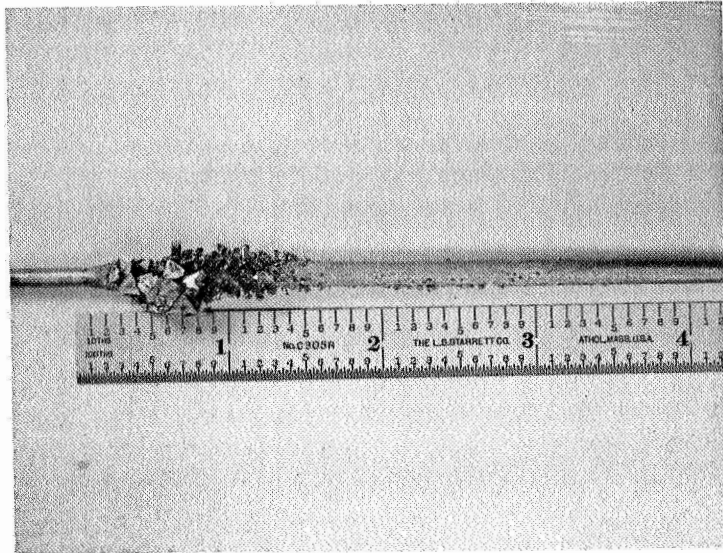


Figure 16. Reactor Design



CRYSTAL GROWTHS
THAT APPEAR ON
UPPER ZONE OF
FILAMENT (EXP. 55)

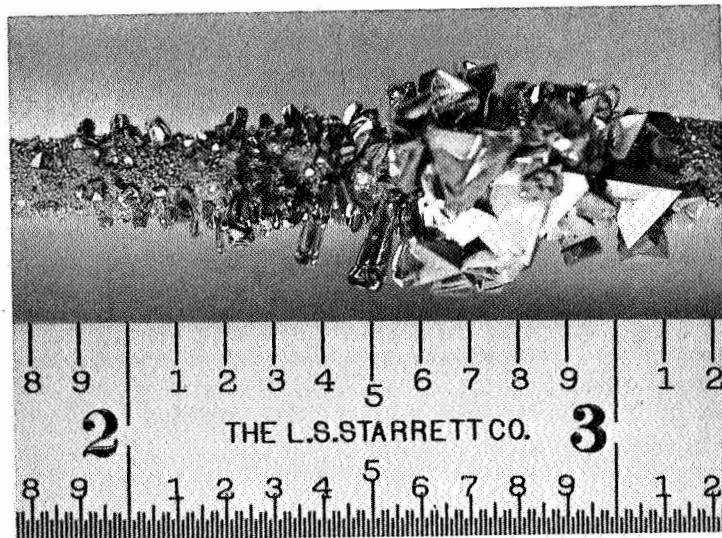
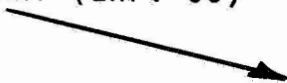
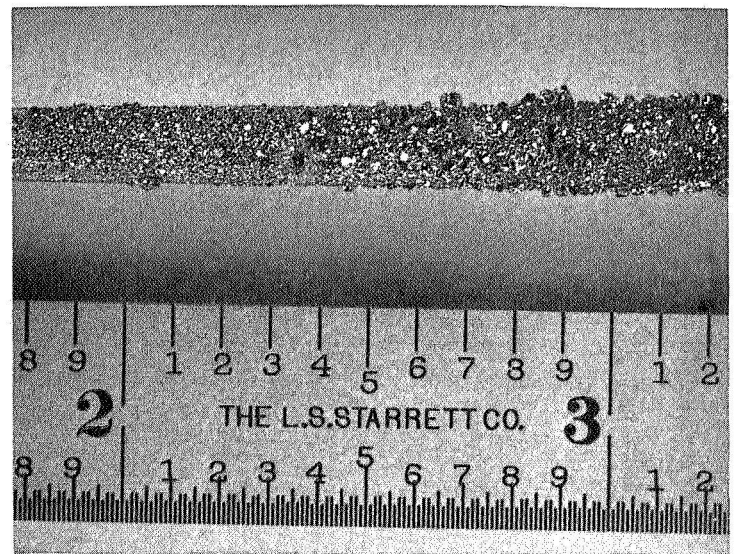
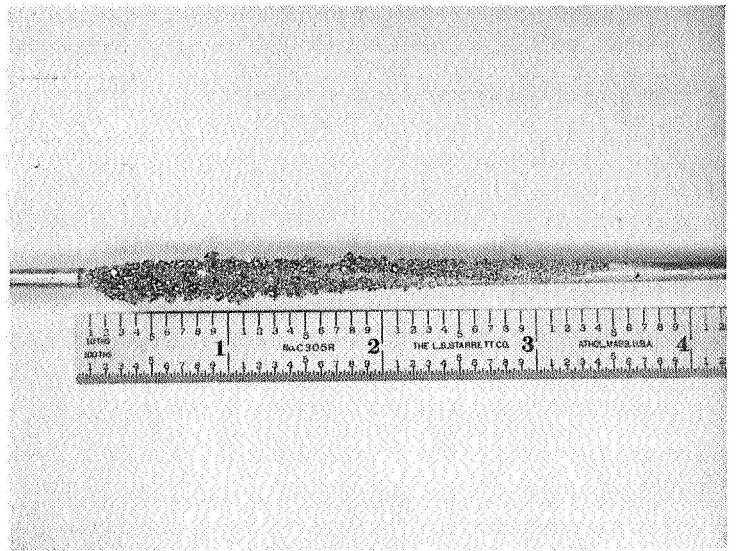


Figure 17. Chromium Deposit, Experiment 55



NEEDLE-LIKE GROWTHS
THAT APPEAR AT UPPER
ZONE OF FILAMENT
(EXPERIMENT 56).

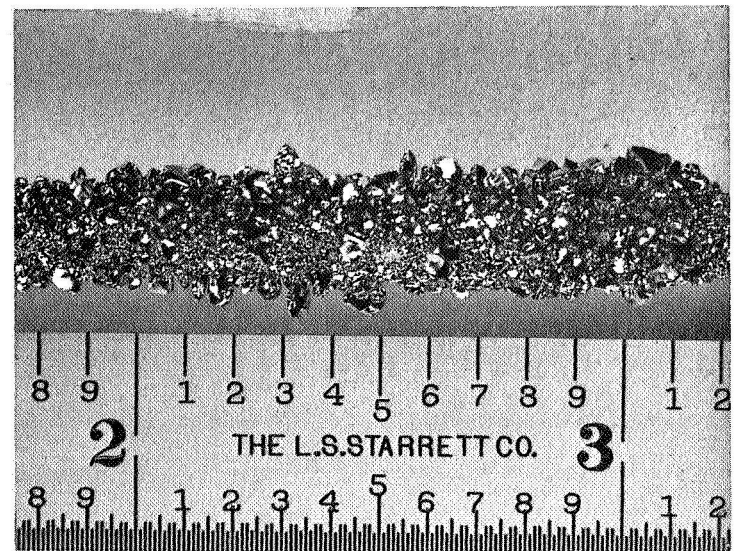


Figure 18. Chromium Deposit, Experiment 56

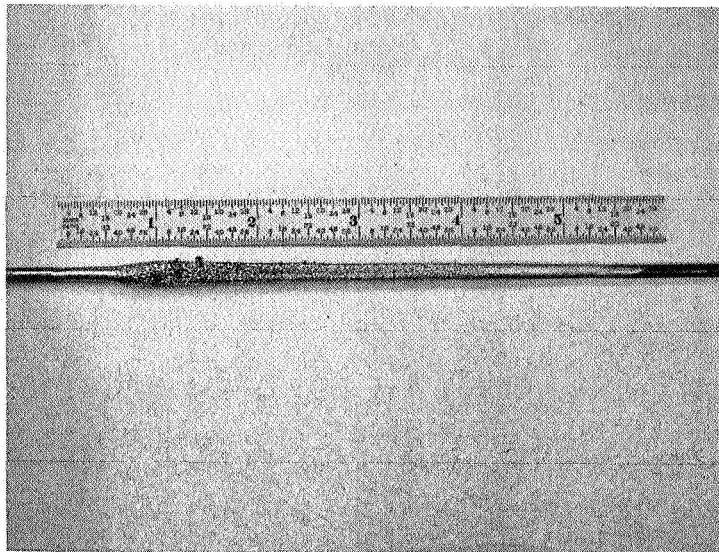


Figure 19. Chromium Deposit, Experiment 69

Experiments 71 through 73 were conducted to observe the effect of HCl mixed with the H_2 flow on the texture of the deposit. As expected, the addition of HCl improved the uniformity and texture of the deposit. Figure 20 is a photograph of experiment 71.

Side experiments were performed to determine the filament temperature profile, since crystal growths were more predominant in that area which was believed to be at a higher temperature. A blank run (without HCl) was made under the usual run conditions, and the filament temperature was measured. Temperatures of $950^{\circ}C$ at the upper part of the filament and $800^{\circ}C$ near the middle were observed. Subsequently, an experiment at a higher deposition temperature yielded a profusion of needle growths. Removal of the HCl which had been added to the hydrogen favored needle or crystal growths of some type. Also noted were crystal growths which were beginning on the substrate of the filament.

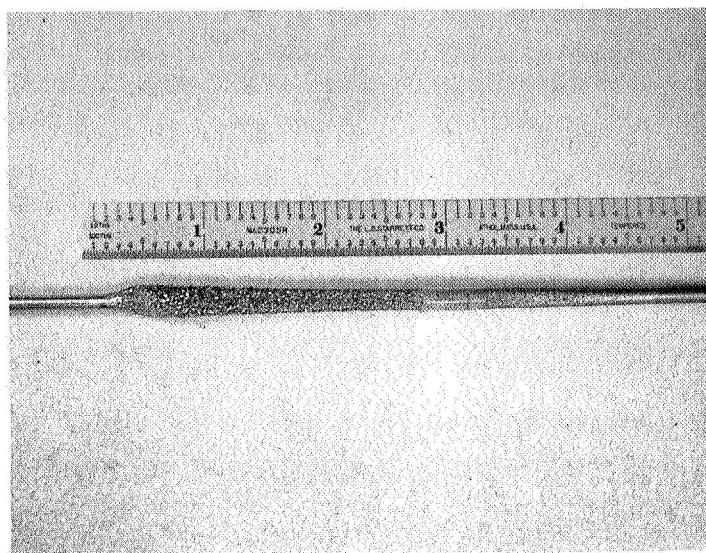


Figure 20. Improved Uniformity and Texture Obtained by Adding HCl to Hydrogen in Experiment 71

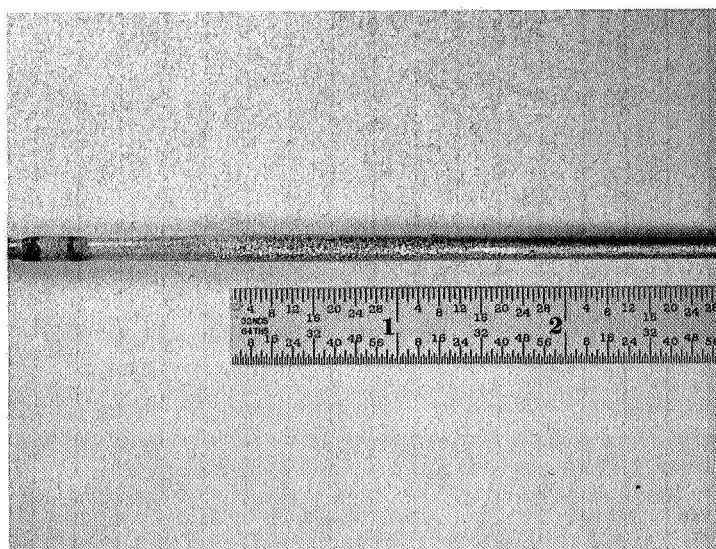


Figure 21. Effect of Lowered Filament Temperature, Experiment 85

Experiments 76 through 78 were conducted primarily to test the effect of the dispersoid on the uniformity of the deposit. The addition of the dispersoid to the substrate of the depositing chromium was expected to favor a more uniform polycrystalline coating by providing continuous nucleation sites for crystal growth. Al_2O_3 (0.1μ) was fed into the chamber through the H_2 feed line at a rate of 18 mg/min. Needle growths continued to appear as before; the conclusion was that adding the dispersoid had little or no effect on the texture of the deposit, at least in these experiments.

Since needle growth was observed to have begun on the filament's substrate, several surface preparations were employed in experiments 79 through 83.

- The surface of the filament was sandblasted with little or no influence on needle growth, since sporadic growths appeared
- A sandblasted substrate and a physically vapor-deposited chromium film covering were applied so that the vapor-deposited coating would be started on a smooth chromium substrate rather than on a tungsten substrate where needle growths might be favored; sporadic needle growths were still predominant

In two subsequent experiments, the surface of the filament was sandblasted and a chemically deposited chromium coating applied; then, the needles that appeared on this filament were removed, the coating sandblasted, and a second coating deposited on the prepared substrate. No significant improvement resulted for either experiment.

Although the ratio of CrCl_2 and H_2 concentrations had been varied over a wide range in previous experiments, three additional experiments were performed with lowered H_2 concentration.

In experiment 83, the H_2 concentration was held lower throughout the entire run; a very smooth needle-free coating resulted. Some larger crystals appeared but, unlike those previously observed, were tightly bound to the filament. In this 3-hr experiment, a smooth but tapered coating was obtained. Figure 21 is a photograph of experiment 85. At these run conditions, a run period of no more than 3 hr could be attained because of exhaust-line plugging. Successive runs on one filament were necessary to obtain a larger deposit.

Experiments 86 through 91 were conducted in an effort to grow deposits of a larger diameter. Experiments 86 and 87 were deposited on the coating from experiment 85. Each experiment was run for approximately 3 hr and the rod inverted in the next experiment to smooth the coating nonuniformity. A photograph of the 0.25-in.-dia rod is presented in Figure 22.

Experiments 88, 89, and 90 were run on three different filaments to obtain a smooth initial-run deposit for a new series of runs. Run 89 was selected, and a run deposit with the filament inverted was made during run 91. Figure 23 shows the appearance of the filament at the conclusion of runs 89 and 91. Filament size was increased to 0.20 in. in dia. This filament was broken when it was being removed from the reactor, so additional run deposits could not be made.

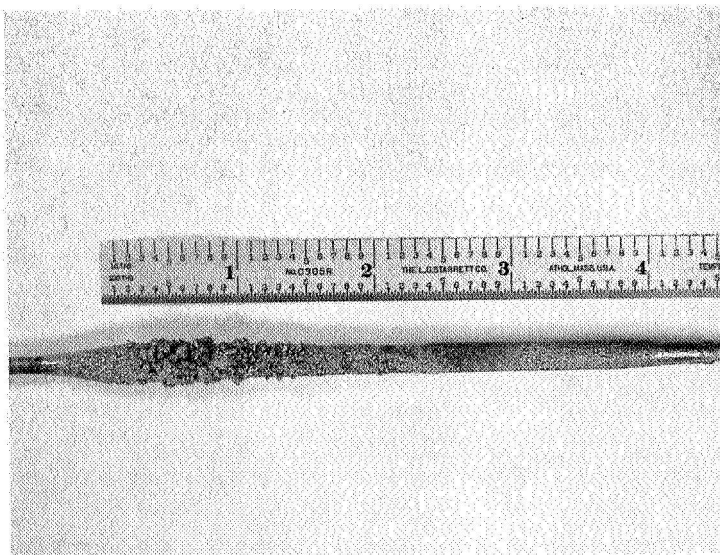


Figure 22. 0.25-In.-Dia Chromium Deposit (Experiments 85, 86, and 87)

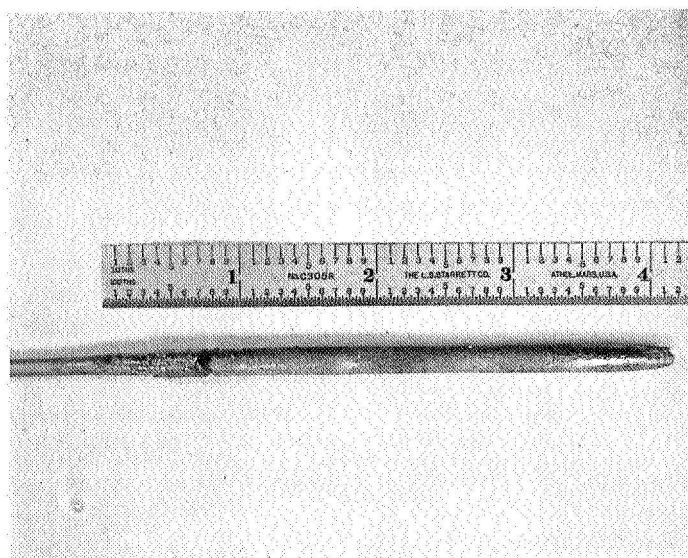


Figure 23. 0.2-In.-Dia Chromium Deposit (Experiments 89 and 91)

V. PHASE III, CHROMIUM THORIUM-OXIDE DISPERSION MATERIAL

The gas system employed in the experiments which introduced the dispersoid into the reaction chamber is shown in Figure 24. There were slight modifications so that a vibrator powder feeder could be used to meter the thorium-oxide powder. Helium, hydrogen, and argon gases (in later experiments) were used to carry the powder to the deposition site.

In initial experiments 92 through 100, only small quantities of thorium oxide could be delivered to the deposition chamber. Packing and plugging in the lines were severe. Rearrangement of the powder delivery lines resulted in a slight improvement in experiments 101 through 109; however, radiochemical analysis of the deposit indicated less than 0.1 percent thorium-oxide content. A low-density or crystalline growth usually appeared on the upper portion of the filament. A typical spongy or granular deposit is shown in Figure 25. To study particle distribution and to evaluate the use of granular material for a test sample, a portion of the material was pressed into a tablet as shown in Figure 26. Surface roughness due to small voids in the tablet prevented the stripping of a replica for electron micrographic examination.

Because of the inability to obtain a chromium thorium-oxide material in the system described in earlier experiments, design changes in the powder-feed system were made, as shown in Figure 27. In this arrangement, thorium-oxide powder was driven down into the deposition chamber via a tube extended through the CrCl_2 generating chamber. Experiments 110 through 130 were conducted in this system with results similar to those with the previous design.

A 1-in.-dia disk, mounted with the surface perpendicular to gas flows, was used in experiments 110 through 129. This provided a larger settling surface area for the thorium-oxide particles.

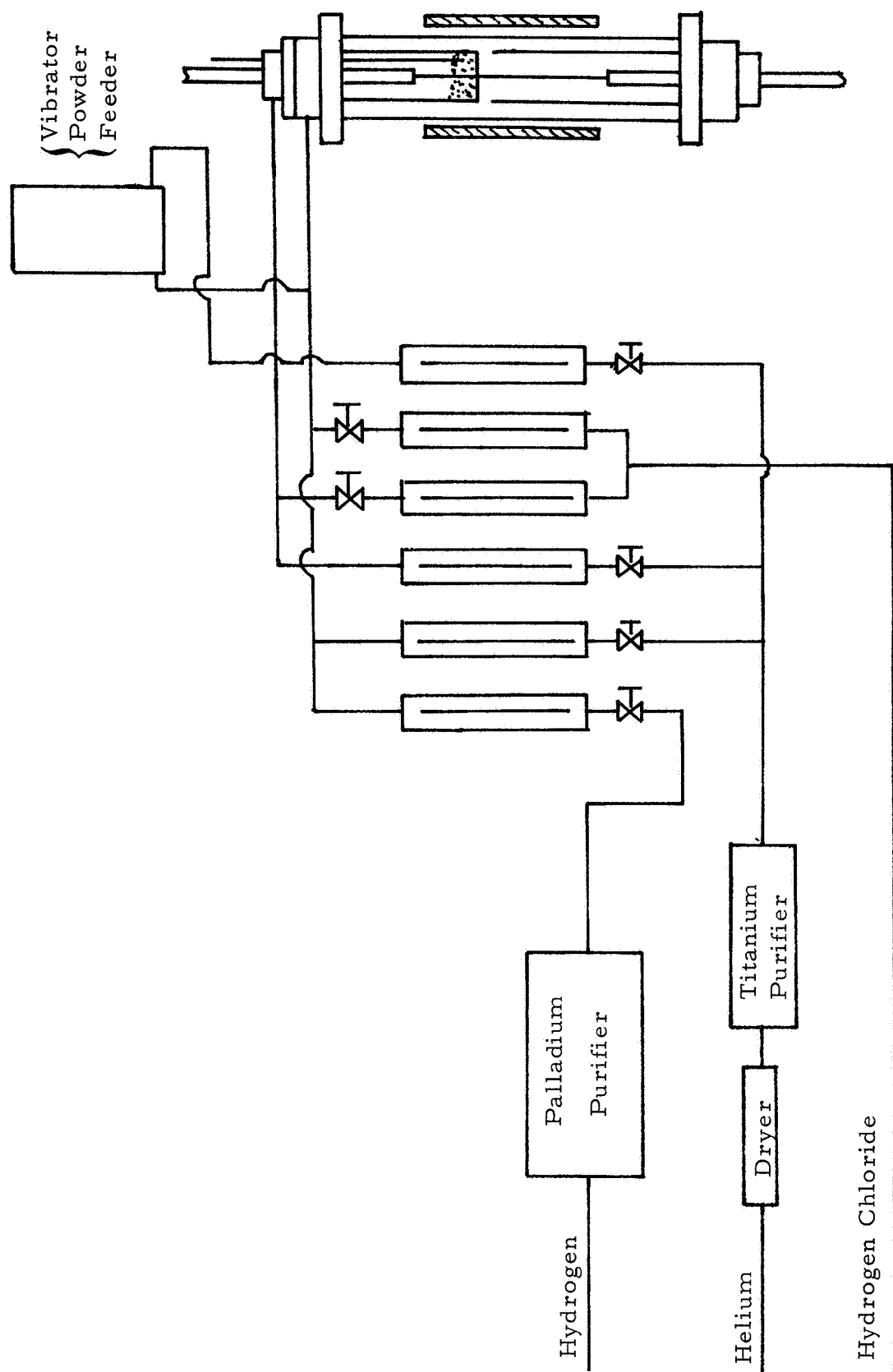


Figure 24. Gas System Used To Introduce Dispersoid into Reaction Chamber

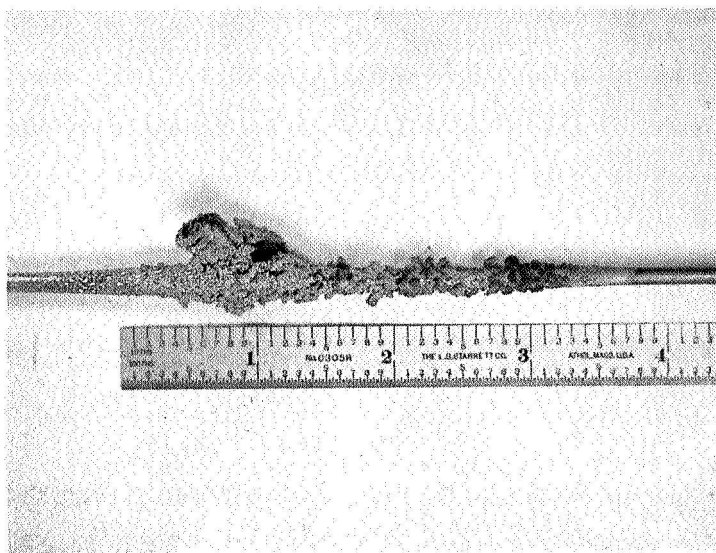


Figure 25. Typical Spongy Chromium Deposits Containing 0.45 V/O of ThO_2 Found on Specimens of Experiments 103 through 113

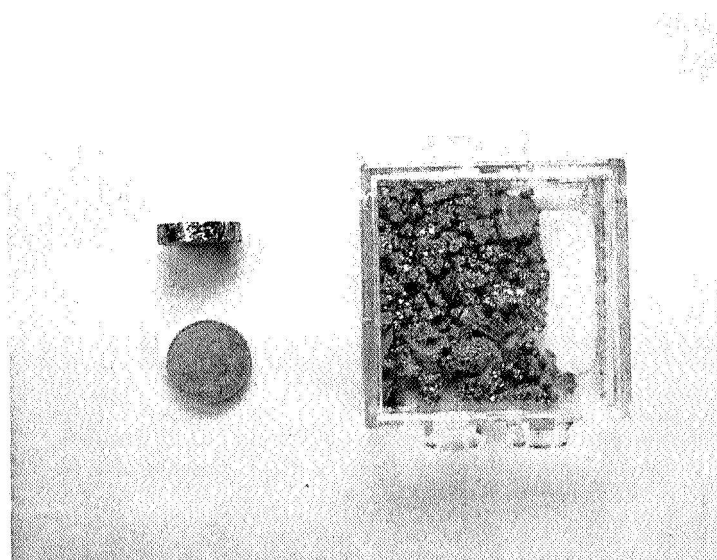


Figure 26. Spongy Chromium Material before and after Pressing into Tablets

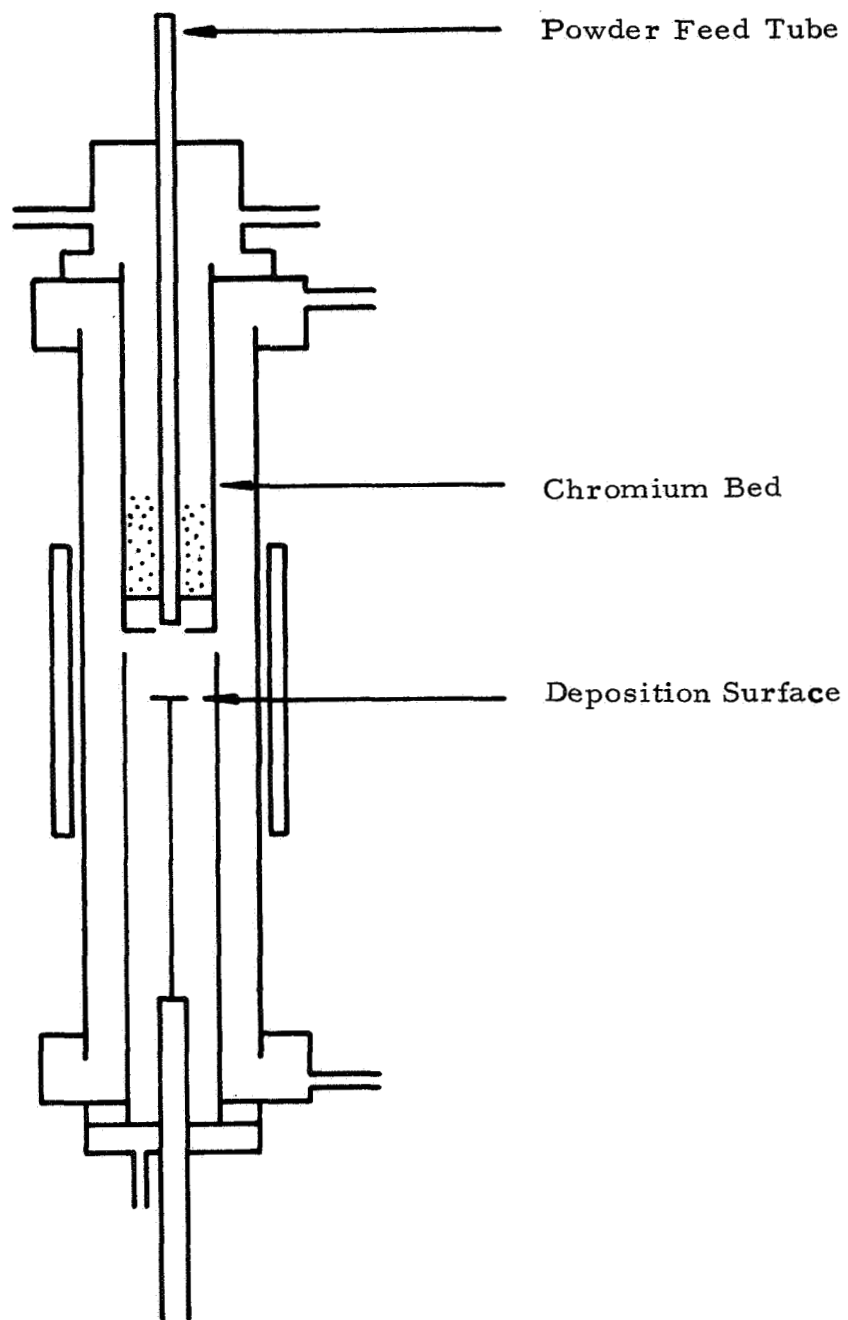


Figure 27. Reactor Design

A typical low-density deposit is shown in Figure 28, and a high-density deposit is shown in Figure 29.

Deposits from experiments 110, 117, and 125, analyzed by wet chemical analysis, contained a larger amount of thorium oxide (as indicated in Table II). The deposit from experiment 117 was used for electron micrographic examination.

In final experiments 130, 131, and 132, a tungsten filament was used to build up a 3/8-in. -dia x 4-in. -long deposit suitable for testing. This sample was sectioned for temperature exposures and electron micrographic examination.

VI. PHASE IV, PRODUCT EVALUATION

A. CHEMISTRY

Representative samples were analyzed for carbon, sulfur, nitrogen, oxygen, and hydrogen. Combustion chromatography was used to determine carbon and sulfur content; vacuum fusion was used to determine nitrogen, oxygen, and hydrogen content.

Emission spectroscopy indicated trace quantities of iron, nickel, and aluminum (Table II).

X-ray analysis of residue from a digested sample showed the presence of ThO_2 and Cr_2O_3 , with additional lines indicating other chromium oxides.

B. 4-SPECIMEN STABILITY TEST

An inert atmosphere furnace was used to perform the 4-specimen stability test. A cross-section of each specimen was metallographically polished, and hardness measurements were performed before and after exposures (Table III). Figure 30 is a typical hardness measurement performed before and after exposure.

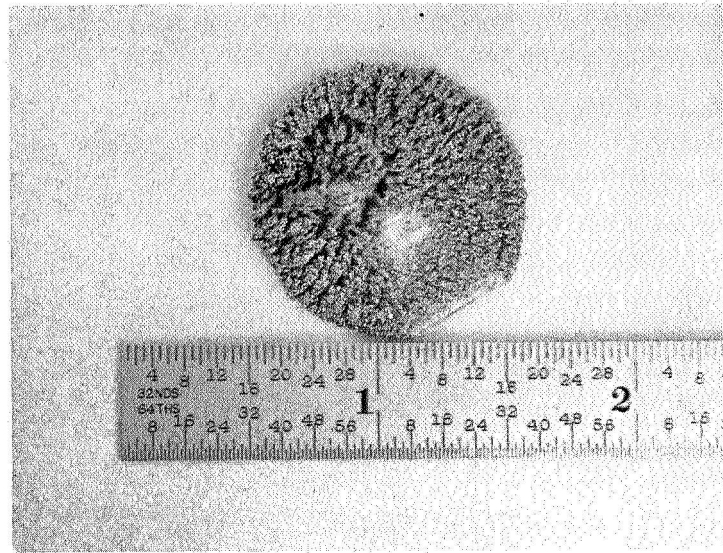


Figure 28. Top View of Disk at Completion of Experiment 121

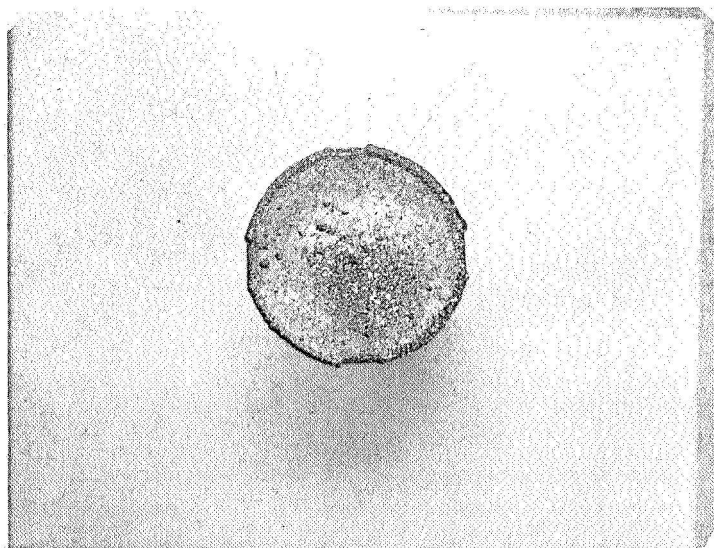


Figure 29. Top View of Disk at Completion of Experiment 115

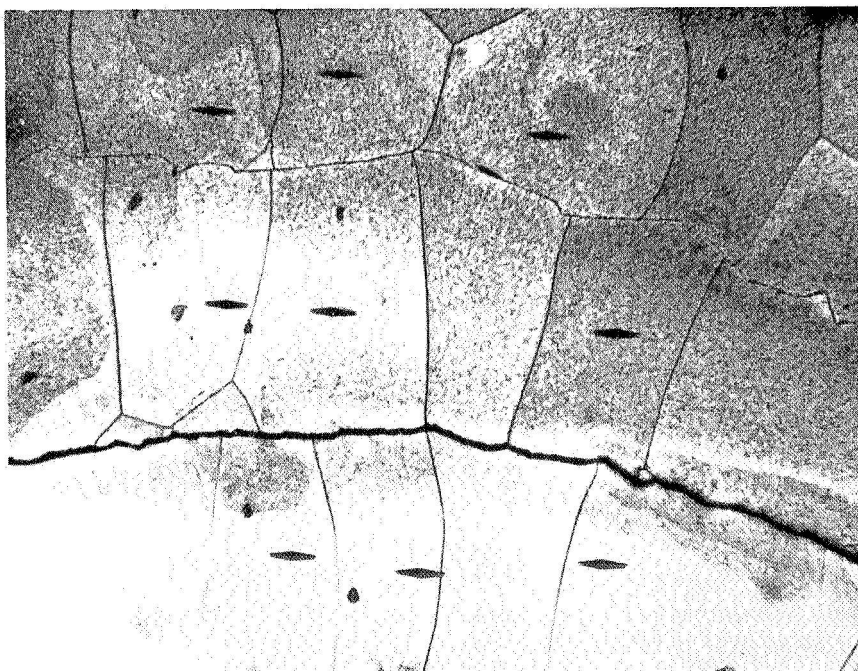
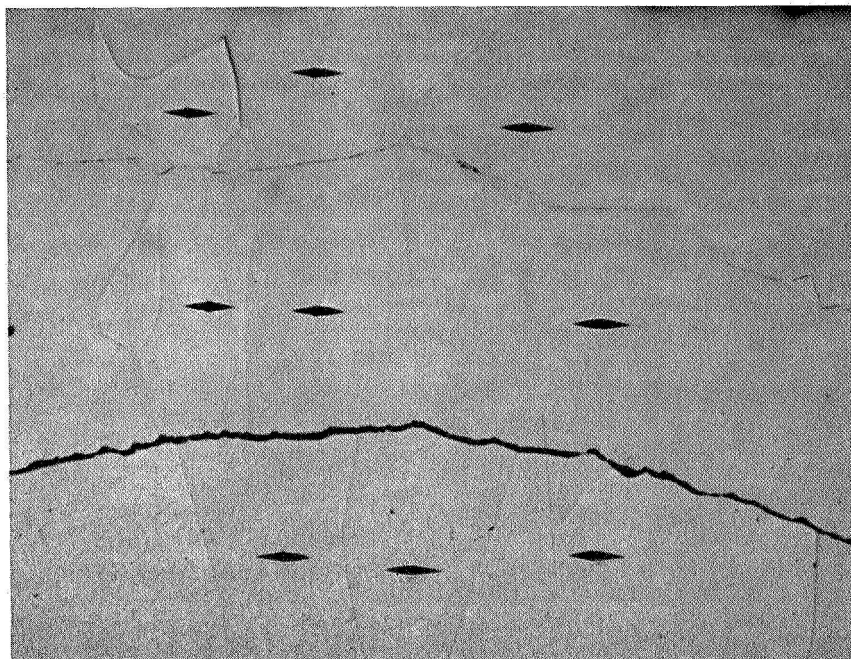


Figure 30. Hardness Measurements before (Top) and after (Bottom) Temperature Exposure (2400°F for 10 hr)

Figure 31 is an electron micrograph of a specimen before exposure showing evidence of dispersoid. Figures 32 and 33 are electron micrographs of the samples after a 100 hr. 2600°F exposure. In Figure 33, the large islands shown appeared to be particles that had migrated into an agglomerate of some material, such as an oxide, which has reacted with the matrix. These areas occurred only at the interface line between successive growth cycles. An electron microprobe analysis of that area of the specimen did not detect evidence of thorium. Some contamination in the interface resulting from start up or shut down in successive runs is evident in photomicrograph shown in Figure 30.

Figure 34 is an electron micrograph of ThO_2 powder used in the program.

C. CONCLUSIONS

The process of chemical vapor deposition has shown feasibility for deposition of pure chromium metal in rod form. Samples to 3/8-in. dia x 4-in. -length were prepared.

The chemical analysis shown in Table 4 indicates that the target-level purity for the chromium metal was obtained for all elements; the purity level of the chromium is corroborated by hardness and room-temperature workability. This purity could be significant in improving long-term dispersoid stability.

Efficiency of the process measured as the ratio of chromium transferred as the chromium chloride to the weight of the chromium deposited on the filament was 11 percent. Deposition rates were ordinarily less than 1.5 gm/hr.

Inclusion of the thoria in the chromium matrix was accomplished; however, gas entrainment, metering the powder uniformly remained a problem throughout the program. A more stable system is needed.

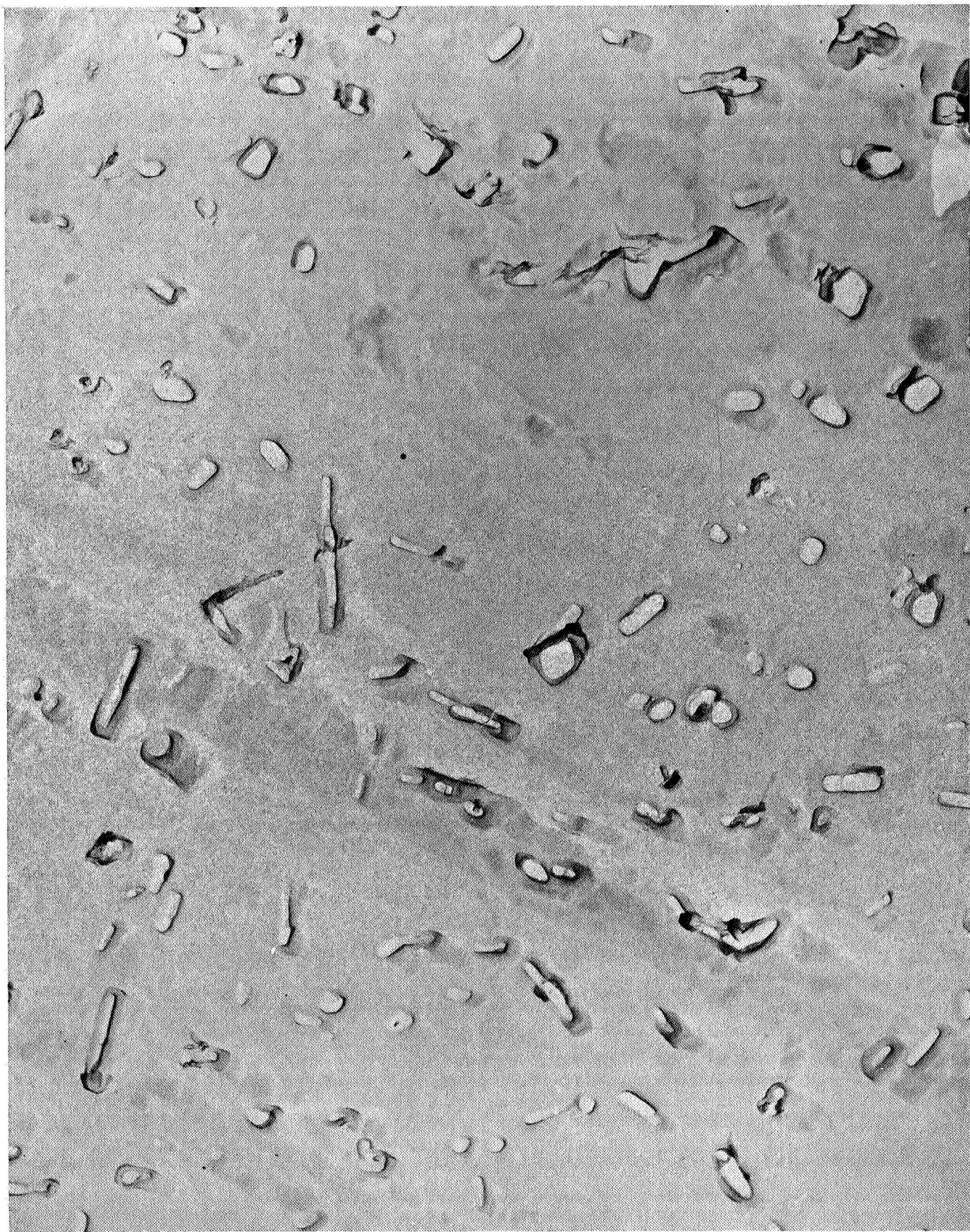


Figure 31. Electron Micrograph of Specimen before Exposure
Showing ThO₂ Particles (11,000 mag)



Figure 32. Electron Micrograph of Specimen after 100-hr, 2600^oF
Exposure(11,000 mag)

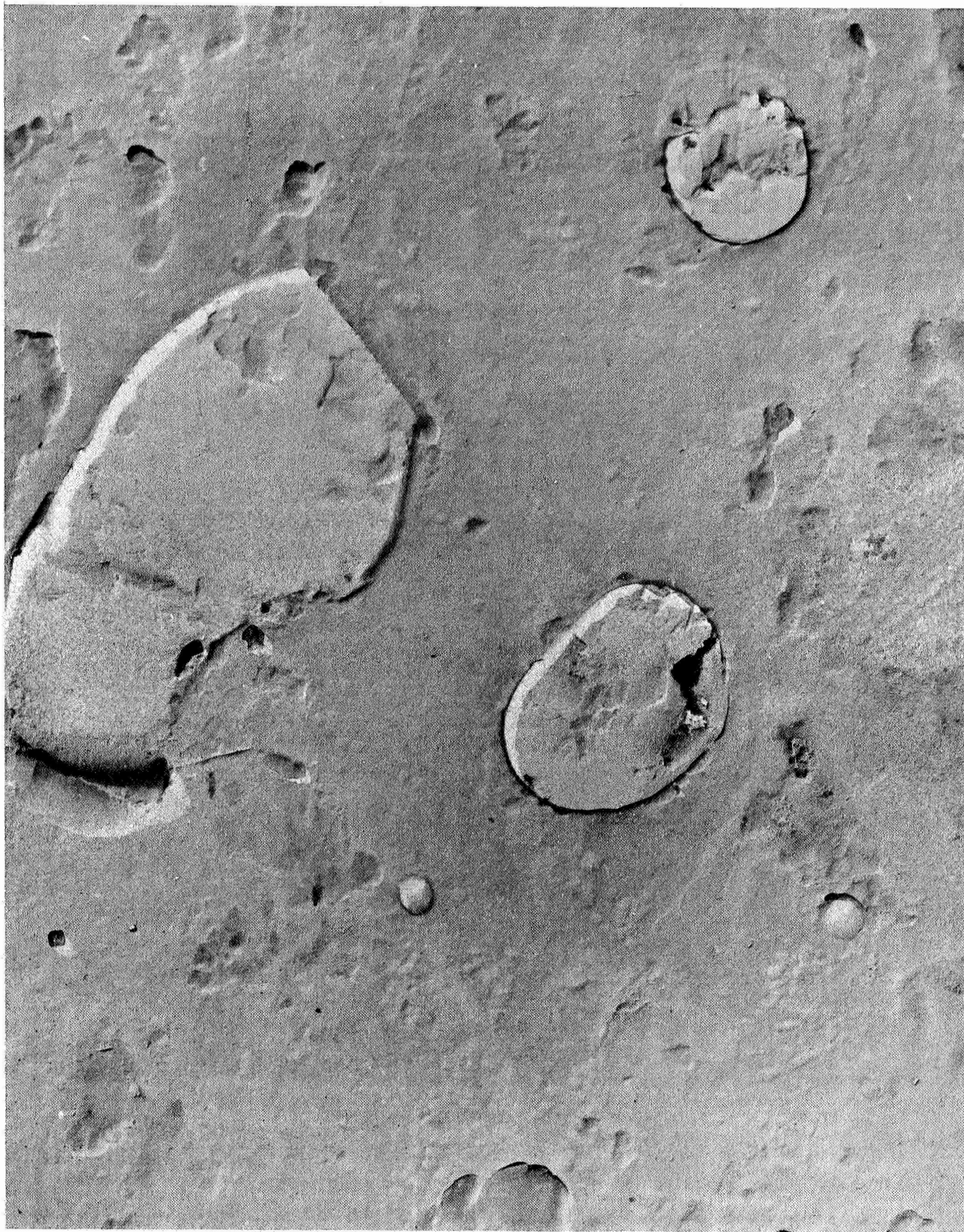


Figure 33. Electron Micrograph of Contamination in the Interface of the Specimen(11,000 mag)

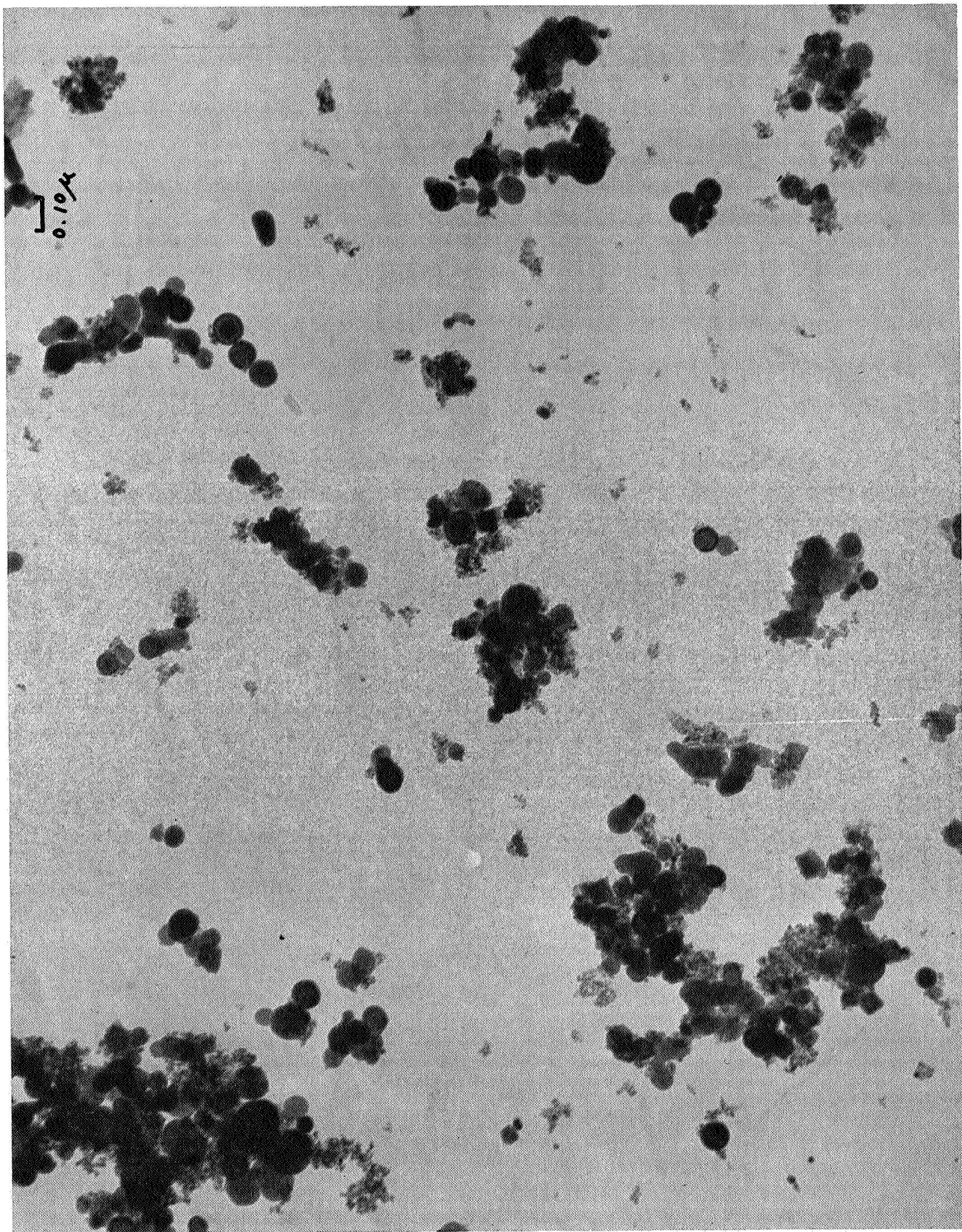


Figure 34. ThO₂ Particles (at 75,000 mag)

The inability to obtain the desired microstructure is attributed mainly to powder-feeding problems. There is no evidence indicating that this process would not be satisfactory in obtaining the microstructure if a solution to the feed problem were obtained.

Maximum thermal treatment of 100 hr at 2400°F and 2600°F resulted in some change in the microstructure of the deposits containing some thoria.

The areas of interaction as depicted in Figure 33 were observed at the "growth rings" caused by stopping and restarting. A green, presumably oxide material, was observed in these areas which was undoubtedly chromium oxide. The electron microprobe data supports this conclusion as little thorium is observed to result from reaction of chromium and chromium oxide during the thermal treatment. These "growth rings" are not inherent in the process but result from the small size of the experimental equipment.

The thermal treatment caused a recrystallization to occur in the samples, and the matrix area away from the "growth ring" was identical in hardness with the unheated material; indicating no interactions had occurred.

D. RECOMMENDATIONS

- Emphasis needs to be placed on obtaining reliable control on the powder to be entrained in the matrix and on methods of delivering the powder to the deposition site.
- The process should be developed so that a sufficient quantity of pure chromium (1- to 2-lb lots) can be produced to permit the study of process economics and efficiency.

Table I
RUN CONDITIONS

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
1	0.4	4.0	-	0.275	1000	950	48	-	-	31.0	-	-	No deposit
2	0.4	6.0	-	2.0	1200	950	60	-	-	19.4	-	-	No deposit
3	0.4	6.0	-	2.0	1200	950	60	0.05	0.83	26.3	-	-	Very light, smooth deposit
4	0.4	4.0	-	2.0	1200	950	55	-	-	24.0	-	-	No deposit
5	0.3	4.0	-	2.0	1200	950	55	0.30	5.4	21.2	-	-	Needle growth opposite gas jet; smooth deposit above
6	0.3	6.0	-	2.0	1000	950	150	0.6	4.0	45.5	-	-	Needle growth above a smoother coating
7	0.2	4.0	2.0	2.0	1200	950	60	0.21	3.3	14.8	-	-	Much smoother coating; only slight needle growth

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
8	0.3	6.0	2.0	3.0	1200	950	120	0.30	2.5	34.3	-		Very dark-colored deposit
9	0.3	6.0	-	2.0	1200	950	60	0.10	1.67	18.92	-	Inverted	Light deposit
10	0.1	4.0	-	2.0	1200	1000	60	0.10	1.67	10.80	-	Inverted	Light deposit
11	0.2	8.0	-	2.0	1200	1000	60	0.11	1.67	12.31	-		Very light deposit
12A	0.150	8.0	-	4.0	1200	850	30	-	-	9.0	-		No deposit
12B	0.075	8.0	-	4.0	1200	850	30	-	-	-	-		No deposit
13	0.150	8.0	-	4.0	1200	850	60	-	-	15.7	-		No deposit

* Except in runs 12A, 12B, and 13 which are Cl₂

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
14	0.150	4.0	-	4.0	1200	850	60	-	-	16.77	-	Inverted	No deposit; very little CrCl ₂ made
15	0.150	4.0	-	4.0	1200	900	30	0.15	5.0	15.92	-	Inverted	Deposit of crystals on right wall
16	0.150	4.0	-	4.0	1350**	900	60	-	-	10.47	-	Inverted	No deposit; CrCl ₂ not moved to filament
17	0.150	4.0	-	4.0	1200	900	60	-	-	15.52	-	Inverted	No deposit
18	0.150	4.0	-	4.0	1200	900	60	-	-	-	-	Inverted	No deposit
19	0.150	8.0	-	4.0	1200	850	60	-	-	8.08	-		No deposit
20	0.150	8.0	-	4.0	1200	950	60	-	-	-	-	Right side-up	Slight deposit

* Except in run 20 which is HCl

** Base of filament

Table I (Contd)

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
21	0.150	8.0	-	4.0	-	1000	60	-	-	-	-	Right side-up	No deposit
22	0.300	4.0	-	2.0	1200	920	60	-	-	-	-		No deposit
23	0.400	4.0	-	2.0	1200	1000	90	1.39	19.0	-	-		Deposit in bottom of reactor directly opposite filament
24	0.300	8.0	-	4.0	1200	1000	60	0.71	12.0	-	-		Light deposit
25	0.400	4.0	-	2.0	1200	1000	90	1.39	19.0	41.6	-		Light deposit
26	CrCl ₃ solid	0.5	-	0.35	1200	950 (Furnace)	60	1.5	25	-	-		Light deposit in colder part of filament
27	CrCl ₃	1.0	-	1.0	1200	950	60	0.45	7.5	-	-		Very light deposit

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition**	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
28	0.025	0.5	-	1.0	1200	775	50	-	-	-	-	1	No deposit
29	0.025	0.5	-	1.0	1200	800	90	-	-	-	-	1	No deposit
30	0.05	1.0	-	1.0	1200	950	60	-	-	-	-	1	No deposit
31	0.025	4.0	-	2.5	1200	850	75	-	-	-	-	1	No deposit
32	0.10	4.0	-	2.0	1200	900	45	-	-	-	-		No deposit
33	0.20	4.0	-	2.0	1200	900	60	-	-	-	-		No deposit; Chamber broke
34	0.20	4.0	-	2.0	1200	900	60	-	-	-	-		No deposit

* Except in runs 32, 33, and 34 which are HCl

** Experiments use CrCl₃ as a starting material

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
35	0.3	4.0	-	2.0	1200	950	60	-	-	-	-	-	No deposit
36	0.4	4.0	-	3.0	1200	950	60	2.0	33	27	-	-	Larger, reasonably smooth deposit
37	0.2	4.0	-	3.0	1200	950	60	0.6	10	8	-	-	Light smooth coating over 3- to 4-inch zone
38	0.3	2.0	-	3.0	1200	950	60	-	-	-	-	-	No deposit; HCl misdirected in chamber
39	0.3	2.0	-	3.0	1200	950	60	3.05	58	20	-	-	Very rough, dark-colored deposit
40	0.15	1.5	-	2.0	1200	950	60	0.30	5.0	16.0	-	-	Very light, smooth deposit
41	0.30	2.0	-	2.0	1200	950	60	-	-	-	-	-	No deposit

Table I (Contd)

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
42	0.30	2.0	-	3.0	1200	950	60	2.0	33.0	29.0	-	-	Rough bulk deposit
43	0.30	2.0	-	3.0	1200	950	60	2.0	33.0	29.0	-	-	Rough bulk deposit
44	0.40	4.0	-	3.0	1200	950	60	0.70	11.0	29.0	-	-	Light, reasonably smooth deposit
45	0.40	4.0	-	3.0	1200	950	45	0.15	3.3	28.0	-	-	Light, smooth deposit
46	0.30	3.0	1.0	3.0	1200	950	45	0.20	4.0	19.0	-	-	Light, smooth deposit
47	0.30	3.0	-	4.0	1200	950	60	2.85	44.0	29	-	-	Rough, dark-colored deposit
48	0.25	2.0	-	4.0	1200	900	60	1.50	25.0	15	-	-	Light, dark-colored deposit

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (°C)	Chromium (°C)							
49	0.20	2.0	-	4.0	1200	1000	60	0.05	0.83	10	-	-	Light deposit concentrated on upper region of filament
50	0.25	2.0	-	4.0	-	1000	60	1.55	26.0	15	-	-	Light, crystalline deposit
51	0.25	2.0	-	4.0	-	1000	60	1.75	29.0	17	-	-	Crystalline deposit
52	0.25	2.0	-	3.0	1200	1000	60	0.9	15.0	14	-	-	Crystalline in upper region; smooth in lower
53	0.25	3.0	-	3.0	1200	950	60	1.05	17.0	15	-	-	Reasonably smooth, crystalline deposit in upper region
54	0.25	3.0	-	3.0	-	1000	60	1.2	20.0	21.0	-	-	Very smooth, uniformly distributed deposit
55	0.25	3.0	-	3.0	-	1000	180	7.2	40	42	-	-	Profusion of crystals in upper region

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (°C)							
56	0.25	3	-	3	65	1000	180	4.5	25	49	-	-	Needle growth in upper region of filament
57	0.25	4.0	1	3.0	65	1000	180	3.75	21	44	-	-	Crystalline upper region; smooth lower region
58	0.25	3.0	-	3.0	65	1000	180	4.05	23	37	-	-	Smother, with dis-colored appearance
59	0.25	3.0	-	3.0	75	1000	90	2.55	28	22	-	-	Reasonably smooth
60	0.25	3.0	-	3.0	75	1000	180	3.20	10	32	-	-	Smooth but tapered deposit
61	0.33	4.0	-	4.0	75	1000	285	12	42	92	-	-	Large crystals at top but smooth and uniform in lower region
62	0.25	4.0	-	4.0	75	1000	300	11.0	37	75	-	-	Crystals at top, with smooth lower region

Table I (Contd)

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight OF Deposit (GMS)	Rate OF Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (°C)							
63	0.33	4.0	-	4.8	75	1000	210	10.85	50	70			Crystalline top; smooth lower region
64	0.33	4.0	-	4.0	75	1000	180	1.65	7	67	-	-	Light but smooth deposit
65	0.33	4.0	-	4.8	75	1000	180	6.0	33	60	-	-	Smooth, discolored deposit
66	0.25	4.0	-	4.0	75	1000	180	4.7	26	65	-	-	Smooth, discolored deposit
67	0.29	4.0	-	4.0	75	1000	360	13.1	36	70	-	-	Needle growths over most of deposit
68	0.225	4.5	-	4.5	75	1000	240	7.8	30	50			Crystal growths over most of deposit
69	0.33	4.5	-	4.5	75	1000	210	7.3	35	65			Smooth, sporadic crystals

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight OF Deposit (GMS)	Rate OF Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition*	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (°C)							
70	0.37	4.5	-	4.5	75	1000	180	6.5	36	60	-	-	Large crystal at top; smooth lower region
71	0.33	4.5	0.033	4.5	75	1000	180	7.55	42	55	-	1	Smooth but tapered coating
72	0.33	4.5	0.015	4.5	75	1000	180	5.7	32	55	-	1	Smooth deposit but sporadic crystal growth
73	0.33	4.5	0.033	4.5	85	1000	180	6.2	34	55	-	1	Smooth lower portion; large crystal in upper portion
74	0.33	4.5	-	4.5	75	1000	180	6.75	38	62	-	-	Sporadic crystal growths; deposit discolored
75	0.33	4.5	-	4.5	100	1000	180	6.55	36	50	-	-	Profusion of crystals
76	0.33	4.5	-	4.5	75	1000	120	3.0	25	45	-	2	Discolored appearance; small crystals

- * 1. HCl added to H₂ flow
2. Dispersoid added at 18 mg/min

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight OF Deposit (GMS)	Rate OF Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition*	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (°C)							
77	0.33	4.5	-	4.5	75	1000	180	6.2	45	45	-	2	Profusion of crystals
78	0.33	4.5	-	4.5	50	1000	30	1	33	15	-	1	Smooth deposit
79	0.33	4.5	-	4.5	50	1000	30	1	33	15	-	2	Smooth deposit
80	0.33	4.5	0.033	4.5	50	1000	120	4.65	39	35	-	3	Smooth deposit; sporadic crystals
81	0.33	4.5	0.033	4.5	50	1000		0.35	18	8	-	4	Smooth sporadic crystals
82	0.33	4.5	0.033	4.5	50	1000	60	2.0	33	30	-	5	Sporadic crystals
83	0.33	4.5	0.033	2.0 4.5	50	1000	90	3.65	40	37	-	6	Smooth, but with numerous crystal growths

- * 1. Dispersoid added at 18 mg/min
2. Number 2 and filament sandblasted
3. Number 3 and chromium evaporated (P.V.D.) onto sandblasted filament
4. Sandblasted filament
5. Crystals removed from run 81, sandblasted, and rerun
6. H₂ concentration lowered to 2.0 l/min for 30 min and then returned to 4.5 l/min

Table I (Contd)

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight of Deposit (GMS)	Rate of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ in Chromium Deposit (%)	Special Run Condition*	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (*C)							
84	0.33	4.5	0.033	2.0	50	1000	90	2.73	30	33	-	-	Very smooth needle-free coating
85	0.33	4.5	0.033	2.0	50	1000	180	5.22	29	60	-	-	Very smooth, clean coating
86	0.33	4.5	0.033	2.0	75	1000	180	5.55	31	55	-	1	Crystals on top; smooth lower portion
87	0.33	4.5	0.033	2.0	50	1000	150	3.85	26	50	-	1	Crystals smoothed out
88	0.33	4.5	0.033	2.0	50	1000	150	3.40	23	50	-	-	Smooth with sporadic crystals
89	0.33	4.5	0.033	2.0	50	1000	180	5.70	32	60	-	-	Reasonably smooth and clear
90	0.33	4.5	0.033	2.0	50	1000	180	4.53	21	60	-	-	Sporadic crystals

* 1. Filament inverted and rerun over previous run

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight of Deposit (GMS)	Rate of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ in Chromium Deposit (%)	Special Run Condition*	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (*C)							
91	0.33	4.5	0.033	2.0	50	1000	180	4.72	26	60	-	3	Dull appearance
92	0.330	4.5	0.033	2.0	50	1000	130	3.72	27	50	-	2	Dull upper region; bright lower region
93	0.33	4.5	0.033	2.0	50	1000	60	1.0	17	15	-	1	Small deposit; sporadic crystals
94	0.33	4.5	0.033	2.0	50	1000	135	5.25	39	45	0.009	1,2	Numerous crystals on top portion
95	0.33	4.5	0.033	2.0	50	1000	150	5.75	38	50	-	1	Numerous crystals
96	0.33	4.5	0.033	2.0	50	1000	240	10.05	42	75	-	1	Clean deposit; no trace of ThO ₂
97	0.33	4.5	0.033	2.0	36	1000	240	8.7	36	75	0.012	1	Crystals in lower portion

* 1. Dispersoid (submicron ThO₂) added
 2. Two L/min of helium added to H₂ flow
 3. Filament inverted and rerun over previous run

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₃ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition #	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (°C)							
	HCl	He	HCl	H ₂									
98					Run failed; helium flow lost							1	
99	0.33	4.5	0.033	1.0	36	1000	180	3.8	21	-	0.083	1,2	Rough nodes
100	0.33	4.5	0.033	2.0	36	1000	180	5.9	33	55	0.001	-	Slight crystal growth
101	0.33	4.5	0.033	2.0	50	1000	180	6.72	37	60	-	-	Numerous crystals
102	0.33	4.5	0.033	2.0	50	1000	120	Run failed; broken tube				1	No deposit
103	0.33	4.5	0.033	2.0	0	1000	180	7.20	40	65	0.45	1	Spongy, low-density material
104	0.33	4.5	0.033	2.0	36	1000	120	4.20	35	40	0.02	1	Spongy, low-density deposit

* 1. Dispersoid (submicron ThO₂) added
 2. Two L/min of helium added to H₂ flow

Table I (Contd)

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl_2 (GMS)	ThO_2 In Chromium Deposit (%)	Special Run Condition ^a	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium ($^{\circ}\text{C}$)							
105	0.33	4.5	0.033	2.0	50	1000	120	4.25	35.4	40	-	4	Spongy, low-density deposit
106	0.33	4.5	0.033	2.0	50	1000	120	4.15	34.8	40	-	1,2	Smooth deposit
107	0.33	4.5	0.033	2.0	50	1000	90	2.9	32	35	-	1	Smooth deposit; powder did not feed
108	0.33	4.5	0.033	2.0	50	1000	60	1.65	27	25	-	1	Spongy, low-density deposit
109	0.33	4.5	0.033	2.0	50	1000	195	7.95	44	65	0.18	1	Spongy, low-density deposit
110	0.33	4.5	0.033	2.0	-	1000	180	5.9	36	45	3.13	1,3	Spongy, low-density deposit
111	0.33	4.5	0.033	2.0	-	1000	180	-	-	35	-	1,3	No deposit

- ^a 1. 1 ℓ /min of Ar added to powder feed
 2. No ThO_2 used in this experiment
 3. 1-in dia disk used as deposition site
 4. Dispersoid (submicron ThO_2) added

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl_2 (GMS)	ThO_2 In Chromium Deposit (%)	Special Run Condition ^a	DEPOSIT DESCRIPTION
					Disk ($^{\circ}\text{C}$)	Chromium ($^{\circ}\text{C}$)							
112	0.50	4.5	0.033	4.0	-	1000	180	6.75	37	55	0.18	1,3	Spongy, low-density deposit
113	0.50	4.5	0.033	4.0	-	1000	65	-	-	35	-	1,3,4	Spongy, low-density material
114	0.33	4.5	0.033	2.0	1000	1000	120	3.85	32	40	0.02	1,3	Dense, clean deposit
115	0.33	4.5	0.033	2.0	1000	1000	120	5.6	47	40	-	2,3	Dense, clean deposit
116	0.33	4.5	0.033	2.0	1000	1000	120	7.1	59	40	0.11	1,3	Dense, clean deposit
117	0.33	4.5	0.033	2.0	1000	1000	110	4.3	42	40	4.75	1,3	Dense, clean deposit
118	0.20	2.0	0.033	2.0	1000	1000	130	5.25	40	40	0.034	1,3	Dense, clean deposit

- ^a 1. 1 ℓ /min of Ar
 2. 1/2 ℓ /min of Ar
 3. 1-in dia disk
 4. Al_2O_3 used instead of ThO_2

Table I (Contd)

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition ^a	DEPOSIT DESCRIPTION
					Disk (°C)	Chromium (°C)							
119	0.33	4.5	0.033	2.0	1000	1000	40	-	-	-	-	1,3	No deposit
120	0.33	4.5	0.033	2.0	1000	1000	75	-	-	33	-	1,3,4	ThO ₂ with Cr over top
121	0.33	4.5	0.033	2.0	1000	1000	105	-	-	47	0.88	1,3,4	Agglomerated ThO ₂ and Cr
122	0.33	4.5	0.033	2.0	1000	1000	120	5.8	-	40	-	2,3,4	Dense, clean deposit
123	0.33	4.5	0.033	2.0	1000	1000	120	3.2	-	40	-	1,3,4	Low-density, granular material
124	0.33	4.5	0.033	2.0	1000	1000	135	9.45	-	45	-	2,3,4	Dense, clean deposit
125	0.33	4.5	0.033	2.0	1000	1000	120	6.72	-	-	6.72	3,4,5	Dense, clean deposit

- * 1. 1 ℓ /min Ar used in powder feed
 2. 1/2 ℓ /min Ar used in powder feed
 3. 1-in dia disk used as deposition site
 4. Powder feed changed
 5. 150 cc/min Ar used in powder feed

RUN NUMBER	FLOW RATE (LITERS PER MINUTE)				TEMPERATURE		Run Time (Min)	Weight Of Deposit (GMS)	Rate Of Deposit (MG/Min)	Chromium Converted To CrCl ₂ (GMS)	ThO ₂ In Chromium Deposit (%)	Special Run Condition ^a	DEPOSIT DESCRIPTION
					Filament (Due to Current in Amps)	Chromium (°C)							
126	0.33	4.5	0.033	2.0		1000	120	6.05	50	40	-	1,2	Clean deposit
127	0.33	4.5	0.033	2.0		1000	150	7.75	51	50	-	1,2	Clean deposit
128	0.33	4.5	0.070	2.0		1000	215	8.23	38	45	-	1,2	Rough deposit
129	0.33	4.5	0.033	2.0		1000	180	-	-	60	-	1,2	Clean deposit
130	0.33	4.5	0.033	2.0		1000	180	5.4	30	60	-	3,4,6	Very clean, smooth deposit
131	0.33	4.5	0.033	2.0		1000	180	6.6	36.7	60	-	3,4,5,6	Very clean, smooth deposit
132	0.33	4.5	0.033	2.0		1000	240	10.9	45	75	-	3,4,5,6	Clean, with crystals on the surface

- * 1. Deposition surface is a disk
 2. 200 cc/min Ar powder feed flow
 3. Deposition surface is a filament
 4. 150 cc/min Ar powder feed flow
 5. Filament from previous experiment inverted and rerun
 6. 500 cc/min He added to powder feed flow

Table II
CHEMICAL ANALYSIS

Sample No.	Type of Analysis					
	Combustion (ppm)		Emission Spectroscopy (ppm)	Vacuum Fusion (ppm)		
	C	S		N ₂	O ₂	H ₂
1	209	56	1-10 Fe <1 Al			
2	86	95	5-50 Fe 5-50 Ni <1 Al		ppm	
3				1	170	0.9
4				9	1376	3

Table III
HARDNESS MEASUREMENTS
AFTER TEMPERATURE EXPOSURES

Sample No.	Exposure		Knoop Hardness No. (100 gm/load)	
	Time (hr)	Temperature (°F)	Before Exposure	After Exposure
1	100	2400	145.2	134.1
			169.9	126.7
			136.8	124.3
			159.3	105.7
			131.5	122.0
			134.1	107.6
			134.1	136.8
			136.8	126.7
			134.1	113.5
2	100	2600	131.5	119.8
			136.8	119.8
			134.1	119.8
			136.8	131.5
			126.7	113.5
			168.1	139.5
			139.5	129.1
			156.0	124.3
			136.8	134.1
3	10	2400	139.5	124.3
			134.1	126.7
			134.1	122.0
			129.1	131.5
			157.6	129.1
			168.1	131.5
			157.6	131.5
			151.2	124.3
			139.5	145.2
4	10	2600	139.5	119.8
			145.2	122.0
			122.0	115.5
			156.0	109.5
			117.6	117.6
			145.2	117.6
			148.2	117.6
			139.5	117.6
			139.5	124.3

Table IV
CHEMICAL ANALYSIS AND HARDNESS MEASUREMENTS

Test Speciman	Knoop Hardness No. (100 gm load)	Chemical Analysis (ppm)						
		Combustion Chromatography		Vacuum Fusion			Emission Spectroscopy	
		(Carbon)	(Sulphur)	N ₂	O ₂	H ₂	Fe	Al Ni
1		209	56					
2		86	92					
3				1	170	0.9	1-10	<1 -
4							5-50	<1 5-50
*5				9	1376	3		
4-Speciman Test Samples	120-160							
Chromium Starting Material	736-830							

*Sample contained trace amount of ThO₂

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